

# Chemical & Process Engineering

## Including CORROSION TECHNOLOGY

Vol. 35, No. 4

### CONTENTS

APRIL 1954

<b>TOPICS OF THE MONTH</b>	103	<b>CHEMICAL ENGINEERING STUDIES AND PROCESS DEVELOPMENT</b> by H. Hoog	124
<b>SLURRY PUMPING AND PIPELINE DESIGN</b> by B. G. Williams, A.C.G.I., B.Sc.(Eng.), A.M.I.Mech.E., A.M.I.Chem.E.	107	<b>PLANT AND EQUIPMENT:</b> Switch fuses; Generating nitrogen for metal treatment; Porous ceramic air slide; Automatic bleed unit; Self-cleaning air filter; Clearing away water by suction	125
<b>ACID SPRAYS IN LEAD CHAMBERS</b> by F. Salsas Serra	110		
<b>CHEMICAL ENGINEERING FOR STUDENTS</b> (Book Review by G. G. Haselden)	112		
<b>ORE DRESSING RESEARCH IN INDIA, 1953</b>	113	<b>CORROSION TECHNOLOGY</b> —See separate section	
<b>ANTI-KNOCK COMPOUNDS—ASSOCIATED ETHYL'S NEW PLANTS</b>	115	<b>WORLD NEWS:</b> From Great Britain, Europe, Greece, Norway, France, Italy, Germany, Turkey, Netherlands, West Indies, Venezuela, Philippines, Canada, India, South Africa, United States, Tunisia, Colombia, British Honduras, Chile	127
<b>ADVANCES IN CATALYSIS</b> (Book Review by P. J. Garner, B.A., Ph.D.)	120	<b>MEETINGS</b>	132
<b>WASTAGE OF REFRactories BY SLAG ATTACK: CAUSES AND EFFECTS</b> by D. Dixon	121	<b>FUNDAMENTAL CHEMISTRY</b> (Book Review)	132

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## Topics of the Month

### More chemical engineering in the universities

AN overdue but none the less welcome increase in university facilities for chemical engineering training is in prospect. King's College (Durham University), Newcastle-on-Tyne, recently invited applications for its newly created Chair of Chemical Engineering and hopes to create a vigorous department based on the existing facilities for chemical engineering teaching and research. Nottingham University, as reported elsewhere in this issue, has created a Chair of Chemical Engineering, Edinburgh University has founded a Chair of Chemical Technology and, finally, Manchester University is reported to be actively considering the appointment of a Professor of Chemical Engineering. It is good that our universities are making strong efforts to swell the flow of chemical engineers to industry. It is up to industry to see that they are effectively used. It would be calamitous if in ten years time there was to be a surfeit of chemical engineers. Such a surfeit would be entirely artificial and would be an indictment of British industry's inability to make the fullest use of chemical engineering.

### How industry can help the H.N.C. scheme

MEANWHILE it is distressing to note that one means of training chemical engineers already to hand is not getting the support it deserves. This is the scheme for

courses for Higher National Certificates in chemical engineering which now applies to the whole of the U.K. Only seven colleges run such courses, compared with 150 which run mechanical engineering courses for H.N.C. Furthermore, the numbers enrolled in these courses are so small that unless better support is forthcoming they may well be reduced. In expressing concern at this neglect in its *Quarterly Bulletin*, the Institution of Chemical Engineers points out that the value of the H.N.C. as a qualification is closely related to the practical training undergone by the holder. Conversely, H.N.C. courses will not flourish unless industry provides suitable training or apprenticeship schemes integrated with courses of study.

As an example of what may be done by industry, the training scheme of National Oil Refineries Ltd., Llandarcy, is cited. This company recruits apprentices who have passed the General Certificate of Education, Advanced Level, in chemistry, physics and mathematics and, in their first year, encourages them to complete the ordinary national certificate courses in chemistry and mechanical engineering while receiving instruction at the refinery in laboratory practice and practical experience in the physical testing of petroleum. In the second year of training the apprentices follow the first year of the H.N.C. course in chemical engineering at the Neath Technical College, being released by the firm on two days each week for this purpose. At the

same time they are learning refinery processes at the works and are introduced to unit operations as applied to petroleum refining.

In the third year of apprenticeship while studying the second-year course for the H.N.C., also on two-day release, the apprentices are given simple exercises in heat transfer, fluid flow, combustion, etc., using refinery equipment under normal operation. At the end of the fourth year the apprentice should have completed the H.N.C. course and should be able to carry out a simple heat and material balance on existing refinery units. Courses of study for the final year are still under consideration, but in the refinery the apprentice will have reached the stage where he can be put to work in the technical department under one or other members of the technical staff. By this time he should be eligible as a Graduate Member of the Institution and, having satisfactorily completed the post-H.N.C. year of study, may be exempted from all but the Home paper of the Associate Membership Examination.

#### **British chemicals: buoyant and booming**

THE British chemical industry is booming. Here are five facts to prove it. In the four years, from 1948 to 1952, the industry has:

Increased its capital from £230 million to £406 million.

Raised capital per employee from £1,600 to nearly £2,700.

Increased production by 29% in volume (nearly double the rate of the rest of the industry) and by 60% in value.

Pushed up productivity by 21% by volume and 51% by value.

Raised exports from £88.25 million to £151.7 million in spite of having to serve home needs first.

This remarkable record is documented by the Association of British Chemical Manufacturers in their *Report on the Chemical Industry, 1953*, a supplement to their 1949 report.

At the end of 1948, the total cost of all expansion schemes proposed for the next five years was just over £190 million. In 1953 the comparable figure was about £230 million. The biggest schemes are for alkalis, including chlorine (£31,261,000), heavy organic chemicals including petroleum chemicals (£28,184,000), plastics and synthetic resins (£17,714,000), inorganic acids and salts (£16,051,000) dyestuffs and intermediates (£15,142,000), and so on down to nine schemes for increasing output of pest control chemicals which will cost £362,000.

The industry is making more use of qualified scientists both in production and in research and development, but it could use more. Total employment has increased from 1,41,817 to 1,51,349, and scientific staff from 6,176 to about 7,400.

The overall picture is of a buoyant and progressive industry which, although once again meeting strong competition from the U.S., Germany and Japan, continues to expand and confidently to plan still further expansion, steadily extending its range of products to meet the ever-changing needs of the economies of the world. These changing needs are most notable in heavy chemicals. Britain can no longer expect to sell as many heavy chemicals abroad because it is the aim of most countries to produce these basic materials for themselves. However, the industry is confident of being able to maintain its leading position by means of the many specialities—high-quality fine chemicals, pharmaceuticals, dyestuffs, plastics and synthetic fibres—evolved through the inventive genius and technical skill of those who serve it.

#### **Forty million pounds of phosphorus**

IN this issue we describe a new plant for the production of bromine which is part of a new chemical complex for the manufacture of anti-knock compounds for gasoline. British production of another important chemical element—phosphorus—is being vastly increased by the starting up of another new plant, also on the west coast, but farther south than Anglesey, the site of the bromine plant.

The new factory, at Portishead, Bristol, belongs to Albright & Wilson Ltd., who have been making phosphorus for over a century. It will have a capacity of 40 million lb. p.a., which is more than the whole output of existing phosphorus facilities in Britain, and should meet all our needs for many years to come. This vast increase has been necessitated by the rise of another industry, synthetic detergents, the connection being sodium tripolyphosphate, a derivative of phosphorus and an important ingredient of detergents. Tripolyphosphate will absorb much but not all of Portishead's output. The phosphorus will be shipped to a new tripolyphosphate factory which Albright & Wilson have erected at Kirkby, Liverpool. The output from these two new plants will entirely replace imports of tripolyphosphate from Continental sources which in 1953 cost us £2 million in foreign currency.

The Portishead site on the south side of the Bristol Channel comprises 20 acres adjoining the dock and facing one of the main power stations of the S.W. Electricity Board, so that the new factory will have direct shipping facilities for raw material and an adequate supply of electric power for its furnaces.

Phosphate rock, the main raw material, is shipped principally from North Africa. Because it is fine it is well suited to pneumatic handling and readers may recall the picture of the powdered rock being unloaded by suction gear from a ship's hold which we printed in December (p. 398). This is the first time phosphate rock has been handled in this manner. Another raw material—anthracite—is also unloaded pneumatically. The rock phosphate is fed into eight 100-ft.-high circular storage bins with a combined capacity of 24,000 tons. Anthracite and granite chippings are stored in spaces between the silos.

Handling of these large quantities of materials is quite automatic. All materials are dried before being automatically weighed and mixed. Waste gases from the furnaces are used in the calciners. The mix is conveyor-carried into hoppers above the phosphorus furnaces and from there it is fed in by gravity feed.

There are six electric reduction furnaces, each capable of taking a load of 7,500 kw. Periodically each furnace is tapped and the slag runs out into covered ways; any fumes emitted are extracted by fans and discharged into high chimneys.

The phosphorus vapour mixed with CO is continually drawn off and passes through dust extractors to condensers which condense the vapour to liquid phosphorus and allow the CO to pass through. Condensed phosphorus is stored in heated underground chambers and kept molten. From here it is pumped into special 24-ton rail tankers for shipment to Kirkby. To run this large plant there will be 180 people, of whom 25 will be staff; of the remainder, 60 will be maintenance men.

The factory was designed by Albright & Wilson's engineers, but much of the construction was undertaken by well-known contractors.

## Comical Engineering Terms



### "DESTRUCTIVE TESTING"

#### Petrochemicals safety code

THE advance of petroleum chemistry has resulted in the commercial production of chemicals, such as acetone, previously obtained from other sources, but the intervening chain of complicated chemical reactions places them outside the definition of 'other products of petroleum' in the Petroleum (Consolidation) Act of 1928, since this definition covers only the primary distillation products. But acetone and kindred products are nevertheless highly inflammable and some guidance on their handling is called for. Now, the storage and transport regulations laid down for petroleum spirit are intended for garages, etc., where the primary object is the protection of the public, whereas many of the highly inflammable products not covered by the Act are used for further processing in factories where access by the public does not arise. There is obviously a need in such factories for a safety code to supplement statutory regulations.

This need was recognised some time ago by the Association of British Chemical Manufacturers and, with Governmental blessing, they set to work to draw up a voluntary code of precautions for use with highly inflammable liquids which are not subject to the Petroleum Act. The code has now been published by the A.B.C.M. as Safety Circular 97 and all members are asked to observe it as far as practicable. It has been examined by H.M. Chief Inspector of Explosives and H.M. Chief Inspector of Factories, who agree that it forms a sound basis for precautionary measures in the use and storage of highly inflammable liquids. It has been based to a large extent on the regulations applying to petroleum spirit under the Petroleum Act, but some of the recommendations go beyond the requirements of the official regulations. It should be noted, however, that the code does not detract from the obligation to observe any statutory requirements which may apply under the Petroleum Act, the Factories Act or any other Act.

Clear instructions are laid down in the code on such subjects as the placing, construction and installation of storage and process vessels, as well as their repair and maintenance; the transfer of liquids; the construction and fitting out of buildings; fire fighting and precautions against static electricity, as well as general safety measures where personnel are concerned.

#### Aden refinery ahead of schedule

GOOD progress is being made in the construction of Anglo-Iranian's 5-million-tons/year oil refinery at Aden and it is expected to start production before the end of this year. Since work began in November 1952, more than 135,000 tons of equipment and materials have been shipped to the site, including 117,000 tons from the United Kingdom and 17,000 from the U.S.A. Of this tonnage, 82,000 tons are for permanent work in the refinery and port.

The refinery is described by its designers as comparatively simple. Its main product will be 3 million tons of fuel oil, 2 million tons of which will be pumped to the company's oil bunkers in Aden harbour which, since the loss of Abadan, have been filled from refineries in the U.K. The refinery and the tank farm will cover 275 acres. Besides the distilling towers and plant for improving the quality of the kerosine and motor fuel, the area will contain a power station and pumps which will bring 4 million gal. of water from the sea each hour for cooling.

Construction of the SO<sub>2</sub> plant and the two distillation units is well advanced and the erection of all other process plants has been started. Several major oil storage tanks are complete and 15 more are being erected. At the port, 1 million cu. yd. of rock have been quarried and tipped into position to form a breakwater and bund which are nearing completion. About two-thirds of an estimated 5.7 million cu. yd. of sand have been dredged from the approach channel and harbour and pumped ashore to reclaim 200 acres on which tankage and port buildings are being erected.

#### State-owned chemicals: a counter-attack

ALTHOUGH the nationalisation of chemicals may seem a dormant issue, if Britain has a Labour Government in the next year or so it will become a very live one. Thus there is every justification for Imperial Chemical Industries issuing a reply to the Labour Party's proposals for chemical nationalisation. It is a detailed and reasoned reply, full of facts and figures, in refreshing contrast to the ambiguities and half-truths of Labour's proposals. In a foreword, Dr. Alexander Fleck, chairman of I.C.I., gives six reasons for not nationalising his company, it being quite obvious that the planners would do this as a first step. Rightly, he points out that nationalisation would slow down all major policy decisions, stifle commercial and technical initiative, and damage the company's overseas interests, so dependent on the goodwill of other nationals. Furthermore, research would be upset and staff difficulties would arise.

In the rest of the booklet, which has been issued to all employees and stockholders of I.C.I., each of the arguments for nationalisation is examined and refuted in detail. The implied criticism in the statement 'Chemical production must expand to keep pace with the enlarged requirements of other basic industries' is answered with two facts: (1) since 1945 I.C.I. have spent £146.5 million on new capital construction in the U.K. alone; and (2) by 1953 the company's output was about 80% more than in 1946.

On monopoly, I.C.I.'s reply is that in the chemical field, as in many others, it is inevitable that some firms should specialise in certain products and also that the production of certain chemicals should be concentrated in a small number of firms. It is claimed, however, that 'There are very few' I.C.I. products that are not subject to competition from identical products, suitable alternative products, or from traditional natural materials. Furthermore, there are many important chemicals that I.C.I. does not produce at all.'

In the next paragraphs, I.C.I. switch from the defensive to the offensive and make several telling points against statutory monopoly which, by law, is placed above the sanctions which must operate against private monopoly. For example, 'A private monopoly is always faced with potential competition, and (this) can be just as effective a safeguard as actual competition; a statutory monopoly destroys both' and 'Statutory monopolies are able to tell the consumer to take it or leave it, and have in fact done so.'

Finally, I.C.I. examine the naively optimistic Labour proposal to acquire only certain sections of the industry which admits only some of the obvious difficulties in stating that 'Final decisions as to boundaries of public ownership must rest upon technical and administrative considerations.' It is, of course, the immense complexity of the chemical industry that poses the most formidable problems of nationalisation. Select only some companies and many companies making the same or similar products will remain outside the ring to compete with the nationalised industry. Specify only certain products and all the makers of these products must be brought in, bringing with them a host of products not specified, from baby foods to disinfectants. If an attempt were made to avoid these two sets of difficulties by splitting off from each company the products on a specified list, the chemical industry would be thrown into chaos.

It may be that the sheer complexity of the chemical industry will eventually deter the planners if and when they get down to nationalisation in earnest. But, remembering the line beginning 'Fools rush in . . . it would be unwise to rely on this. We must have an informed public opinion on the chemical industry. Indeed, it would be helpful to have a properly informed Labour Party. I.C.I. have taken the first overdue step in this direction. But it is only the beginning, and collectively and individually the industry must actively preach against nationalisation for the next few critical years at least.

### Cancer in the chromates industry

CHROME ulceration of the skin or of the nasal mucous membrane has for many years been a serious problem in every country where chromates are made or used, but it is only recently that they have been indicated as a probable cause of lung cancer. In the U.S.A., growing suspicion of this hazard led to an extensive enquiry. The results, recently published,\* show an incidence of lung cancer in the 897 chromate workers in the city of Boston 325 times that of the general population at 40 to 49 years.

In Britain the problem of chrome ulceration has been met by statutory control, including periodical medical examination of workers in nearly all branches of the chromate industry. Most of the cases notified now are associated with chrome plating; the notifications from chrome tanning, dyeing and finishing are very few. In 1951 there was an increase in all cases of chrome ulceration over those notified in 1950; this was largely accounted for by 80 cases occurring in the manufacture of bichromates.

Commenting on the U.S. investigation, the *British Medical Journal* points out that there is certainly no such prevalence of lung cancer in the U.K. industry. An enquiry in 1951 among 724 workers in the chromate-producing industry of Great Britain revealed only one case. It was then pointed out that the British process differs from that of both Germany and the U.S.A. in that the residue from the leaching tanks

\*Health of Workers in Chromate-Producing Industry, Federal Security Agency, Public Health Service, U.S.A., 1953.

is not dried and crushed and used as a filler, but is discarded in a moist condition and carried to dumps outside the factory. The residue, when crushed and recirculated as is done in Germany and America, contains certain acid-soluble, water-insoluble chromium compounds which are suspected in the U.S. report, though whether they are directly carcinogenic could not be established. The report recommends further research on the effects of all chromium compounds and, in the meantime, periodic x-ray examinations of all workers in the chromate-producing industry.

### Tonnage oxygen for copper smelting

MANY metallurgical and chemical processes may be transformed within the next generation by the application of oxygen in tonnage volumes. A good example of the economies that can be achieved in this direction is provided by a new unit for producing the vast quantities of oxygen required for the direct flash smelting of copper concentrates, which has been put into operation at Copper Cliff, Ontario, by the International Nickel Co. of Canada Ltd. Inco's new smelting process eliminates the fuel normally required for smelting and makes economical the present large-scale output by Canadian Industries Ltd. of liquid sulphur dioxide from furnace exhaust gases. International Nickel's operations call for a volume of more than 7½ million cu. ft. of oxygen every day—enough to fill 32,000 standard cylinders. This requires the production of 300 tons of 95%-pure oxygen every 24 hr.

The oxygen plant, designed and built by Canadian Liquid Air Co. Ltd. and known as an *Oxyton*, separates the oxygen from atmospheric air by the liquefaction process, using the same basic principles as apply in smaller commercial oxygen plants. The air is first liquefied under pressure in a series of compressors, regenerator-heat exchangers, distillation columns and other equipment, and then the oxygen is separated from the other constituents of the atmosphere: nitrogen, argon, neon and krypton. The oxygen is carried as a gas through a 16-in.-diameter elevated pipeline from the *Oxyton* to the smelter, a distance of 6,000 ft.

As temperatures of a few hundred degrees F. below zero are involved in the liquefaction process, special consideration had to be given to the types of metals and other materials employed in certain parts of the oxygen-generating unit. For example, the important regenerator-heat exchanger system consists of two nitrogen regenerators, each 8 ft. in diameter and 17 ft. long, and two oxygen regenerators, each 4 ft. in diameter and 14½ ft. long. While one pair of regenerators chills the incoming air, the other pair is being chilled by the separator gases. Working temperatures range from 80 to -280°F. Since most ferrous metals suffer a marked increase in brittleness at sub-zero temperatures, the regenerators were made from special 8½% nickel steel developed by Inco for low-temperature use. Incidentally, the regenerators are of welded fabrication, type 310 stainless-steel electrodes being employed.

Two other interesting pieces of equipment in the oxygen plant are a specially built oxygen compressor and a huge turbo-compressor (required for the initial compressing of the air), one of the largest of its kind in the world. Despite its size and intricacies, the operation of the *Oxyton* is essentially automatic once the liquefaction process has been started. Control and metering are carried out by modern electronic equipment with over 40 control instruments centralised on a huge operating panel.

# SLURRY PUMPING AND PIPE LINE DESIGN

By B. G. Williams, A.C.G.I., B.Sc.(Eng.), A.M.I.Mech.E., A.M.I.Chem.E.

Most works engineers at some time or other have to face the problem of pumping a new type of slurry. Without the advantage of past experience in the knowledge of the behaviour of the particular slurry to be pumped, the design of the pipeline and the selection of a suitable type of pump can present a serious problem. The object of this article is to outline a basic procedure which may be adopted in order to obtain sufficient information to make the necessary design calculations.

## Behaviour of slurries

THE coefficient of viscosity for a Newtonian fluid is equal to the tangential force on unit area of either of two horizontal planes, at unit distance apart, required to move one plane at unit relative velocity, i.e.:

$$\mu = \frac{Sxg}{V}$$

where  $\mu$  = coefficient of viscosity,  $S$  = tangential shearing force,  $x$  = distance between planes and  $V$  = velocity of one plane with respect to the other (all in consistent units).

Plasticity is the property of a substance which enables it to be continuously and permanently deformed in any direction without rupture, under a stress exceeding the yield value.

After deformation has started, equal increments of stress will produce equal increments of velocity.

Reverting to the fundamental conception of flow between two parallel planes, since a part of the applied force  $S$  is expended in overcoming the yield value  $S_y$ , the equation for plastic flow becomes:

$$\tau_i = \frac{(S - S_y) X g}{V}$$

where  $\tau_i$  is the coefficient of rigidity of the material analogous to the coefficient of viscosity of a true liquid.

Fig. 1 represents all the recognised types of flow, as follow:

Class 1 represents the flow of a true liquid. The slope of the line is proportional to the coefficient of viscosity.

Class 2 represents the flow of a pseudo plastic material. It can be seen that this curve does not obey the fundamental equation of plastic flow, because the line bends towards the origin at low flow rates. Two examples of pseudo plastics are napalm and cellulose acetate in acetone.

Class 3 represents a true plastic and is a graphical representation of the equations of plastic flow. The 'apparent' viscosity of the plastic at any point  $A$  on this curve, if measured in the usual way for liquids, is proportional to the slope of the line  $OA$ . It is seen that two different velocities such as  $V_A$  and  $V_B$  on the figure correspond to entirely different viscosity lines  $OA$  and

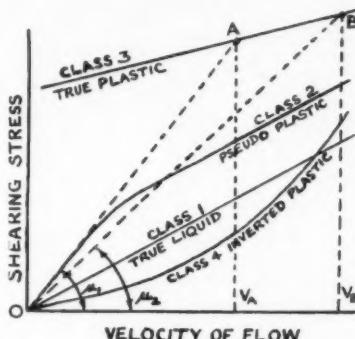


Fig. 1. Graphical representation of all the recognised types of flow.

$OB$ , the slopes of which are proportional to the apparent viscosity. Some examples of this type of plastic are sewage sludge and grain suspensions in water. Thick industrial slurries usually fall into this class.

Class 4 represents the flow of an inverted plastic substance. This material is thin at low flow rates, but becomes increasingly thicker as the rate of shear increases. Some examples of this type of plastic are starch suspensions in water, mica suspensions in water, quicksand and beach-sand.

## Theory

A complete mathematical analysis has been presented by E. C. Bingham in his book, 'Fluidity and Plasticity.'

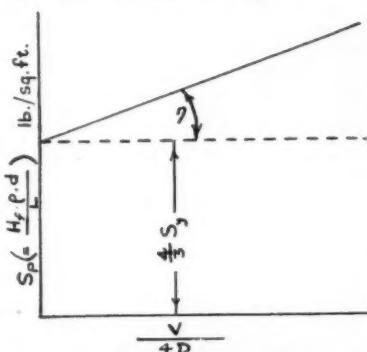


Fig. 2. Graphical method for the determination of  $S_y$  and  $\tau_i$ .

The essential relationships may be abstracted as follows:

$$\frac{H_f}{L} = \frac{16S_y}{3\rho D} + \frac{\tau_i V}{\rho D^2} \quad (1)$$

$$S_p = \frac{4}{3}S_y + \frac{\tau_i V}{4D} \quad (2)$$

$$S_p = \frac{H_f \cdot \rho \cdot D}{4L} \quad (3)$$

where  $H_f$  = friction head loss (ft. of slurry),  $L$  = length of main (ft.),  $S_y$  = shearing stress at the yield point of the material (lb./sq.ft.),  $\rho$  = density of material (lb./cu.ft.),  $D$  = pipe diameter (ft.),  $\tau_i$  = coefficient of rigidity (lb./ft./sec.),  $V$  = velocity of flow (ft./sec.) and  $S_p$  = shearing stress in the flowing material at the pipe wall (lb./sq.ft.).

From these equations it can be seen that  $S_p$  and  $\tau_i$  may be evaluated by direct measurement of the velocity of flow ( $V$ ) of the fluid and the frictional head loss ( $H_f$ ) along a known length ( $L$ ) of pipe of known diameter ( $D$ ).

If a graph is plotted with  $S_p$  as ordinate and  $V/4D$  as abscissa the slope of the resulting line will represent the coefficient of rigidity ( $\tau_i$ ) and the intercept on the ordinate will give  $(S_p = \frac{4}{3}S_y)$  (See Fig. 2).

By substituting these values of  $S_y$  and  $\tau_i$  in equation (1) the plastic flow friction head loss at any velocity in a pipe of diameter  $D$  and length  $L$  may be calculated for any particular slurry.

The turbulent friction loss head may be calculated by normal methods, as the head loss at a given velocity, measured in feet of flowing fluid, will be the same for all fluids and may therefore be deduced from observations made on water, brine, etc. Past observers have not noticed any change in the values of  $S_y$  and  $\tau_i$  when evaluating from experimental readings taken on pipes of various diameters.

## Pilot layout

In order to obtain values of  $S_y$  and  $\tau_i$  for the particular slurry to be pumped, a small pilot pumping system is necessary. The main may be chosen from any suitable

stock and the length should be as long as conveniently possible. The material of the test main should be the same as the final installation. The pressure drop may be measured by means of a differential mercury manometer, fitted with suitable purging connections in order to prevent the blocking of the pressure gauge lines. The rate of flow can be conveniently measured by timing the discharge of the slurry into a tank of known capacity.

The discharge from the measuring tank can be controlled by a suitably weighted wood plug.

A simple arrangement is shown in Fig. 3. Preferably the centrifugal pump should be driven by a D.C. motor in order to obtain varying velocities of flow. With an A.C. motor-driven pump variation in the velocity of flow must be obtained by means of a gagged delivery valve. This is not ideal arrangement, as 'sifting up' will occur in front of the valve from small flow rates.

#### Critical velocity

The critical velocity of the slurry will vary with the pipe diameter, as shown in Fig. 4, although very little variation will be observed for pipe diameters above 3 or 4 in.

The critical velocity may be determined approximately in the following manner.

For plastic flow conditions the friction head loss is given by equation (1):

$$H_f = \frac{16S_y}{3\mu D} + \frac{\eta V^2}{\rho D^2}$$

In turbulent flow conditions the friction head loss is given by:

$$H_f = f \cdot \frac{L}{D} \cdot \frac{V^2}{2g}$$

These two equations may be solved for the critical velocity ( $V_c$ ) giving:

$$V_c = 32\eta + 32\sqrt{\eta^2 + f \frac{S_y \cdot D^2}{96}} \quad (4)$$

The friction factor  $f$  varies with velocity, so that equation (4) must be solved by trial and error, first assuming a value of  $f$ , then obtaining from the friction factor chart the value of  $f$  for the velocity found in the first approximation.

The second value of  $f$  is then substituted in equation (4) and the critical velocity found. Usually one approximation is sufficient.

The critical velocity can be determined from the experimental results obtained from the pilot pumping system by plotting friction head loss against velocity of flow on a log-log basis (Fig. 5).

It must be remembered that this plot gives a critical velocity for the particular slurry under test in a pipe of known diameter. The critical velocity will vary with pipe diameter.

The determination of the critical velocity is important for several reasons:

- It is the most economic pumping velocity.
- Sedimentation is likely to take place

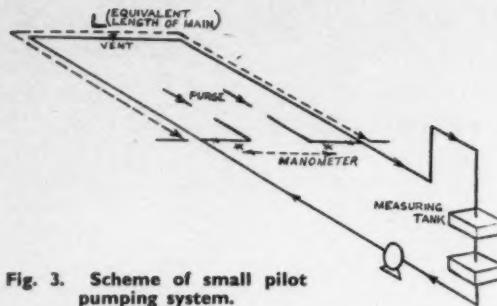


Fig. 3. Scheme of small pilot pumping system.

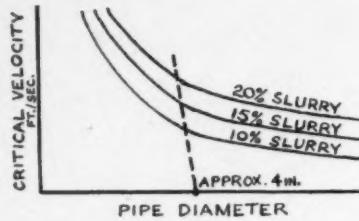


Fig. 4. Variation of critical velocity with pipe diameter.

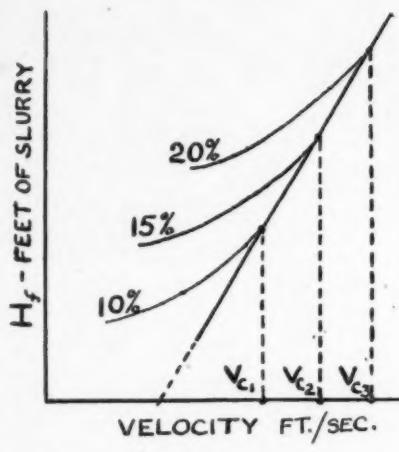


Fig. 5. Plot of friction head loss against velocity of flow.

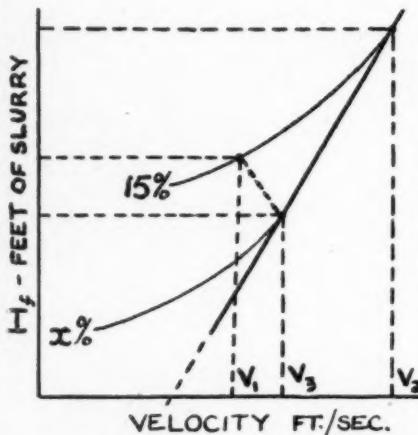


Fig. 6. Reduction in pumping head possible by controlled dilution.

at velocities of flow below the critical.

(c) For a given size pipe it indicates the optimum slurry concentration. Consider a 15% slurry (composition of solids by weight) which would flow at a velocity  $V_1$  in an installed pipe of diameter  $D$ .

This velocity is below the critical velocity ( $V_c$ ). By dilution to a value  $X\%$ , which may be calculated, not only can the velocity of flow be adjusted slightly above the critical velocity, but also the friction head loss will decrease (Fig. 6).

If sedimentation does occur and the sediment is not allowed to remain on the bottom of the pipe for long periods, it is the author's experience that the sediment will be re-entrained by increasing the velocity of flow.

An actual settling velocity may be obtained, without risk of blocking the pilot main, in the following manner.

A taper is introduced into the system and the rate of flow adjusted to a value slightly above the critical. Settling will take place in the taper until the velocity through the free area of flow reaches the maximum settling velocity. After a convenient period the main may be broken and a direct measurement made of the free area of flow, hence the settling velocity may be calculated from measured throughputs (See Fig. 7).

#### Thixotropy

Thixotropy is exhibited by some gels in that they become fluid when subjected to mechanical agitation. The change is reversible.

Thick industrial slurries usually exhibit thixotropy. The mechanical agitation of the slurry in a stock tank generally improves the flow properties, so that thick slurries can be fed into the suction branch of a centrifugal pump.

#### Viscosity and Reynolds number

It has been found that when evaluating Reynolds number,  $\frac{DV\rho}{\mu}$ , if the absolute viscosity of the dispersion medium is used as  $\mu$ , the friction chart obtained by plotting friction factor  $f$  against Reynolds number correlates results for most industrial slurries.

Further, in turbulent flow conditions for most industrial slurries where water is the dispersion medium, the viscosity of the slurry is practically the same as that for water. This is to be expected, as when fully turbulent flow conditions are reached the losses are then due to impact which depend on velocity and density only, and are independent of viscosity.

#### Reynolds-Moody chart

Using the absolute viscosity of the

dispersion medium, say water, a consistent plot of friction factor  $f$  against Reynolds number is obtained.

When these results are plotted on the Reynolds-Moody chart (Fig. 8), the plastic flow region becomes immediately clear and an 'effective' absolute viscosity of the slurry under plastic conditions of flow can be inferred so as to place the plastic flow portion of the curve on the laminar flow region of the Reynolds-Moody chart.

It will be noticed that the observed plastic flow line is not parallel to the laminar flow line  $AB$ , being closer at the bottom than at the top.

This fact is explained by reference to Fig. 1. It will be seen that, as the velocity of flow increases, the 'apparent' viscosity decreases. Consequently the adjustment to be made to the viscosity of the slurry (assumed to be that of the dispersion medium) becomes increasingly less as the region of turbulent flow is approached.

This is an extremely simple method of obtaining values of the 'apparent' absolute viscosity, and for the original plot any value for the absolute viscosity of the slurry may be used, although it is wiser to use the value for the absolute viscosity of the dispersion medium, as correlation for turbulent rates of flow may be obtained.

The effective absolute viscosity may be checked by another method. Assuming Poiseuilles law applies at the critical velocity, then

$$P = \frac{32\mu LV_c}{gd^2}$$

where  $P$  = pressure drop in lb./sq.ft.,  $\mu$  = absolute viscosity in lb./sec./sq.ft.,  $V_c$  = critical velocity in ft./sec.,  $L$  = length of main in ft. and  $D$  = diameter of pipe in ft.

Substitute  $H_f$  (friction head loss in ft. of slurry) for  $P$ , then

$$H_f = \frac{P}{\rho}$$

$$\mu = \frac{H_f \rho d^2}{LV_c}$$

By substituting known values  $H_f$ ,  $\rho$ ,  $L$  and  $V_c$  the absolute viscosity may be found.



Fig. 7. Method of determining settling velocities.

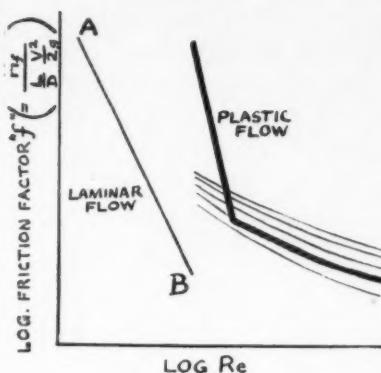


Fig. 8. Plot of friction factor  $f$  against  $Re$ , showing region of plastic flow.

### Application of the results

From the test results it is possible to construct friction head loss-velocity curves for the diameters of pipe most likely to be used on the proposed disposal system. From this information it is possible to select the economic diameter of pipe both from the point of view of capital cost and annual pumping costs. The flexibility of the pumping system must also be considered, i.e. the fluctuation in quantities of solids to be disposed. In this respect, if the pipe is suitable for the thickest possible slurry, when the solids to be disposed of decrease temporarily, the slurry may be diluted in order to ensure that the velocity of flow is always above the critical.

Under no circumstances should results obtained from one type of slurry be applied to another type purely on the basis of solid concentration. In the author's experience, slurries of the same solid concentration and with only slightly different chemical compositions may give

friction head losses that vary by 300%, together with large differences in critical velocities.

### Pumps

Centrifugal pumps may be safely used up to pumping pressures of 300 p.s.i. Good suction arrangements must be provided to prevent cavitation in the eye of the impeller. Materials of construction for the impeller, neck and rings must be carefully chosen to withstand abrasive wear should the slurry be of an abrasive nature. A gland-sealing fluid should be supplied to prevent abrasive wear on the pump sleeves. This fluid should be carefully controlled to prevent unnecessary dilution of the slurry being pumped. Arrangements should be made to flush the pump out immediately slurry pumping ceases. Casing thickness should be adequate to withstand erosion by the slurry in the volute chamber.

The pump should be chosen so that the speed of rotation is kept as far as possible below 1,100 r.p.m. This will ensure longer pump life and less pump maintenance.

### Summary of procedure when pumping a new slurry

The following procedure should be adopted when handling a new slurry:

(1) The characteristics of the slurry are determined by pressure drop experiments in a test main, as far as possible with slurries of varying solids concentration.

(2) The values of  $S_y$  and  $\eta$  are found by

plotting  $S_p = \frac{H_f \rho D}{4L}$  (equation 3) against velocity of flow, as illustrated in Fig. 2.

(3) These values of  $S_y$  and  $\eta$  are substituted in equation 1 ( $\frac{H_f}{L} = \frac{16S_y}{3\rho D} + \frac{\eta V}{\rho D^2}$ ).

Assuming various pipe diameters and a known pipe length  $L$ ,  $H_f$  may be calculated for various velocities of flow.

(4) These values are plotted as illustrated in Fig. 9.

(5) Knowing the amount of solids to be disposed of, the economic slurry concentration, rate of flow and pipe diameter may be determined.

(6) Knowing the pressure drop and the quantity of slurry to be handled, a suitable pump may be selected.

(7) The method of dealing with a pump failure must then be considered in order to prevent slurry standing in the pipeline.

Amongst the possibilities are:

- The installation of a spare pump.
- The installation of a spare pump—steam driven.
- The coupling of a high-pressure water main to wash the slurry along the pipe.

(d) Drain connections at suitable low points in the main.

The final decision will depend upon the plant design and requirements.

(Reading list on page 114)

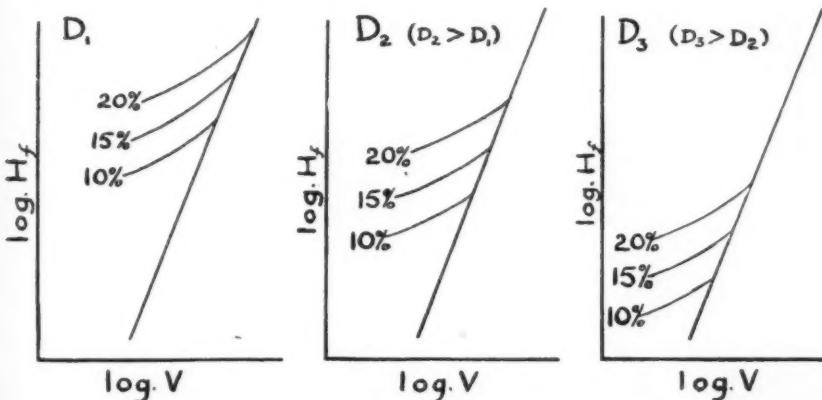


Fig. 9. Method of plotting calculated values of  $S_y$  and  $\eta$ . For pipes of varying diameters, showing decrease in friction head loss and critical velocity as diameter of pipe increases.

# Acid Sprays in Lead Chambers

## SPANISH DEVELOPMENT IN THE LEAD CHAMBER SULPHURIC ACID PROCESS NOW BEING USED IN ITALY AND FRANCE

By F. Salsas Serra

*Increased production of sulphuric acid by the lead chamber process is claimed for a method whereby an acid spray is used in the chamber instead of a water spray. The technique has been used in Spain for over 30 years, but it is only recently that it has attracted interest in other countries. Now it is being used in France and Italy. Here is a description of the acid spray process as presented at the technical meetings of the International Superphosphate Manufacturers' Association held in Cambridge, England, last September.*

THE production of sulphuric acid with the aid of oxides of nitrogen—the lead chamber process—has undergone successive improvements aiming at an increase in production per unit of volume of reaction chambers (chambers, towers, etc.).

One of the most important improvements has been the use of a sulphuric acid spray in the lead chamber in place of the water spray. The spraying of sulphuric acid was suggested earlier by experts, particularly Raschig, who saw in this method a means of avoiding the reversion of nitric oxides to an unoxidisable state. In Spain especially it has been developed on an industrial scale since 1920, according to two methods with widely different characteristics.

(a) The Gaillard process, which, after having been established in Spain, was used in many other countries. It is characterised by the use of turbo-dispersers, in cylindrical lead reaction chambers, which project on to the walls sulphuric acid of a practically constant composition and of which a small quantity is dispersed by the sudden impact on the wall.

(b) The process of the firm, S. A. Cros, which, under the direction of Ferdinand Martin, was adopted by that company in 1922, where it has been progressively applied in about 30 lead chamber systems. This process is characterised by the use of centrifugal dispersion apparatus in the chambers in the form of cylinders or parallelepipeds, which produce directly a sulphuric acid mist of sufficient fineness, the composition of which is different in each chamber.

In Spain there are now only a few Gaillard installations, most of the installations, which now belong to S. A. Cros and its affiliated company, Productos Químicos Ibericos, having been adapted to use the new process.

Outside Spain this process had been very little developed until four years ago, when it was adopted in Italy for four lead chamber plants and for two Mills-Packard plants.

An unusual application of the sulphuric

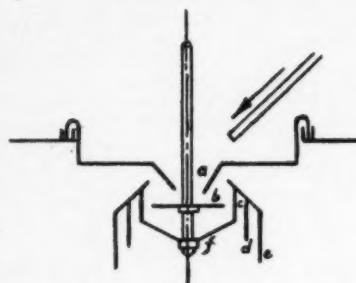


Fig. 1. An early form of centrifugal atomiser which is still used in Spanish sulphuric acid plants. For full description see text.

acid spray occurred in the works at Lérida of S. A. Cros, which I managed from 1927 to 1936.

In this works there were used, as reaction chambers, granite towers originally intended for the manufacture of nitric acid by the arc process. These towers, of decagonal section, have been fitted with lead tops and half emptied of their Silex fillings, the acid being distributed partly by centrifugal sprays and partly by stationary dispersers directly above the filling.

When applying the Kachkaroff process in France for the intensive manufacture of sulphuric acid, we have so utilised and improved the acid sprays in the steel reaction chambers as to permit a great reduction in the volume of the circulation acid, to simplify installations and to diminish initial costs of construction. Of

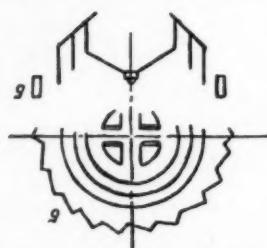


Fig. 2. Stationary sprocket wheel added to the centrifugal atomiser illustrated in Fig. 1 to obtain additional atomising of the acid.

the four installations functioning in France, two employ acid sprays in the reaction chambers, most of which are empty or only partly filled. The same thing applies to the new installations either functioning or in course of erection (seven in Spain and two in Italy).

Finally, in Cerea (Italy) we have applied, for the first time, a combination of the lead chamber and the steel reaction chamber with acid sprays, thereby trebling the production capacity, with very low installation costs and very interesting results from the point of view of production, cost of manufacture and stability.

The conception and the improvement of this technique are based on two factors: (1) Progressive improvement of acid sprays and (2) increased knowledge of optimum conditions of the chemical process.

### Centrifugal atomisers

The first experiments with acid sprays in lead chambers were conducted with the Kestner atomiser used in washing superphosphate gases. We became aware immediately of the necessity to increase the fineness of the droplets, and to avoid a large proportion of the acid being projected on to the walls of the chambers. As  $\text{SO}_2$  is oxidised principally in the liquid phase, every increase in surface should be favourable.

The first apparatus for centrifugal atomisation which we installed, and which is still employed in the Spanish installations, is shown in the form of a diagram (Fig. 1). The acid enters through the cone-shaped hopper *a*, impinges on the disc *b* and is successively propelled on to the surfaces *c*, *d* and *e* forming the bell, which is joined to the axis of rotation by the wheel *f*. The maximum volume which can be conveniently atomised by means of this apparatus is 500 litres/hr.

Later, in order to obtain an additional atomising of the acid projected by the last bell, we added a stationary sprocket wheel, arranged in such a fashion as to produce, by an orthogonal incidence of the acid, the annulation of its driving force and a more pronounced atomisation of one por-

tion of the acid (Fig. 2). This device was employed as from 1927 in the installation of Lérida, where the partial filling of the towers was able to receive without difficulty the acid which had not been atomised by the last impact. The second type of centrifugal atomiser (Fig. 3) is used in the Italian installations of lead chambers and in two intensive installations in France.

The acid enters through the hopper *a* and impinges on the disc *b* and on the discs *c* and *d*, after having been partially atomised on the surfaces by impact at an orthogonal angle *e*. The acid not atomised by the impact on the surface falls on to the radial grooves of the discs *c* and *d*, where it is submitted to a new impact which completes the atomisation of the liquid. The maximum volume which can be completely atomised by this apparatus is 1,500 litres/hr.

The third type of centrifugal atomiser (Fig. 4) was put into service in October 1952 in a works for the intensive manufacture of sulphuric acid at La Rochelle-Pallice; it is also being employed in the two intensive installations at Cerea and Mantova (Italy). The same type will be used in the other installations which are ready to operate or which are in course of construction.

The acid, coming through distributor *a* and nozzles *b*, arrives at a certain pressure on a series of superimposed and identical discs where it hits small blades, the form and angle of which result in a complete atomisation. With this apparatus, the size of which is similar to the others, *i.e.* 25 to 30 cm. in diameter, it is possible to atomise up to 5,000 litres/hr.

The mist obtained with this apparatus is of a remarkable fineness and homogeneity. The use of this apparatus for the distribution of acid in partially filled towers (denitration tower and first reaction tower), as well as in the empty towers (reaction towers or Gay Lussac towers), foreshadows the simplification of installations and an increase in their specific production.

A single apparatus for atomisation according to the same principle will replace with advantage several of the atomisers of the other types in the lead chambers.

#### Composition of atomised acid

Our process differs from the Gaillard process not only in the functioning and efficiency of the atomiser but also because the atomised acid in the lead chambers has a variable composition. During the operation of the chambers it had been constantly observed that a satisfactory functioning occurred within certain limits of density of the acid produced, limits which varied in each chamber and depending also on the composition of the gas, the nitrous supply, exterior temperature and so on. To put it very plainly, one could say that there was either an excess or a lack of water. In comparatively forced production the most frequent disturbance

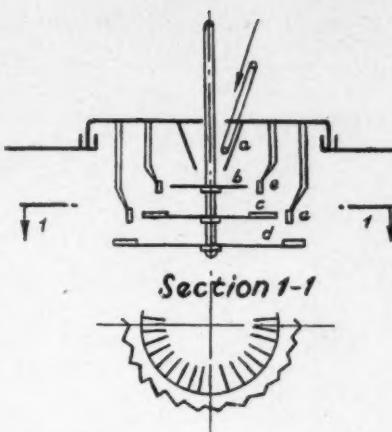


Fig. 3. A centrifugal atomiser used in plants erected in Italy and France. It is capable of atomising 1,500 litres/hr. of acid. For full description see text.

is characterised by an excess of water in the gaseous phase and by a lack of water in the liquid phase. A decrease in exterior temperature rapidly readjusted the situation.

It is therefore understandable that the atomisation of acid, in supporting in each chamber the reaction in the liquid phase as far as volume, density and temperature are concerned, should not only increase the production capacity per cubic metre (thanks to the elimination of calories) but also considerably improve the functioning by rendering it more regular and stable.

The appropriate density of the acid, which is different in each chamber, is below certain dangerous concentrations which result in considerable losses of oxides of nitrogen and in an abnormal wear and tear of the lead. It is, however, possible, especially in the second half of the system, to exceed the dangerous concentration, the acid becoming slightly nitric and the loss of nitric oxides and the abnormal attack of the lead disappearing.

The change-over by dilution of a slightly nitric acid, which has an opaline aspect and a yellowish colour, to an almost de-

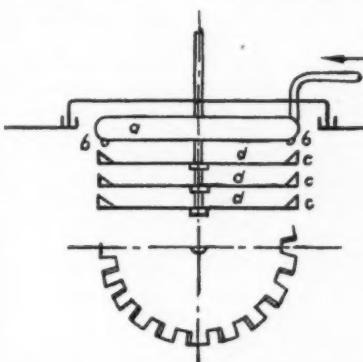


Fig. 4. Up to 5,000 litres/hr. of acid can be atomised with this centrifugal atomiser and a very fine and homogeneous mist is obtained. For full description see text.

nitrated acid which is clear and of rosy colour has an intermediate stage in which the acid bubbles and is the colour of wine-lees. This stage is seen every time that nitrous products decompose; the bubbling is probably due to the generation of  $N_2O$ .

In the course of the experiments, which we made with acids of a density of 55 to 62° Bé and with nitrosity varying over a wide range, which we applied in the towers of the Lérida works, we have encountered the same phenomenon, *i.e.* the appearance of 'dangerous' acid and the considerable loss of nitric oxides every time when, at a given temperature and gas composition, the density of acid was too strong in relation to its nitrosity.

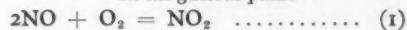
The perfecting of the intensive process, which resulted from the work of Camille Matignon and Pierre Kachkaroff on the simultaneous absorption of sulphurous anhydride and nitric oxides in sulpho-nitric acids, has enabled us to define the relative limits of density and nitrosity of these acids, between which takes place the almost complete oxidation of  $SO_2$  without appreciable loss of nitric oxides, and to avoid at the same time the corrosion of ordinary steel.

It was in 1953, during a course of 'Industrial Catalysts,' organised by the 'Association of Students of the School of Industrial Engineers' in Barcelona, that we postulated, on theoretical grounds, for the first time where this technique could be applied.

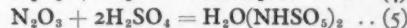
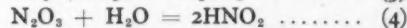
We do not think that the true composition of compounds, which form and decompose in the course of the reactions, resulting in the formation of sulphuric acid, have as yet been defined, but we hope that it will be possible to establish these when applying to the compounds and sulpho-nitrous mixtures the research methods and analyses which have given such good results in the study of sulpho-nitric mixtures.

The theories which we have applied to our technique have already been expounded separately, but we are giving hereunder a summary of the principal ones:

#### In the gaseous phase

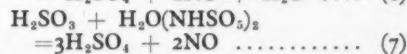
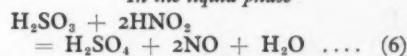


#### In the intermediate phase



Reaction (4) predominates in the lead chambers, where the acid is diluted and slightly nitrous, and reaction (5) predominates in iron reaction chambers, where the acid is concentrated and strongly nitrous.

#### In the liquid phase



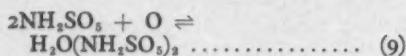
In order to explain the abnormal reactions

which cause a loss of nitric oxides by a reduction to  $N_2O$ , we have assumed a transitory formation of a component, such as  $NH_2SO_5$ , proposed by Raschig and later accepted by Lunge.

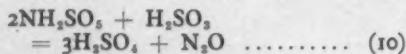
In the diluted and practically denitrated acid this compound would decompose:



If the acid were more concentrated and sufficiently nitrous this compound would oxidise.



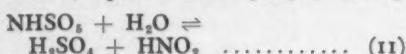
In normal conditions the  $NH_2SO_5$  would react with the sulphurous acid:



giving rise to an irrecoverable loss of oxides of nitrogen

In reactions taking place in iron chambers, and in general in all intensive processes which bring sulphurous gases into close contact with acids of high density and nitrosity, it is indispensable that the oxidising character of the acid be such that the determination of the total content in  $N_2O$  by the nitrometer give a value which is slightly higher (by about 5%) than that obtained by determination with permanganate.

It has been assumed that the sole sulphurous component present in acid is the nitrosylsulphuric acid  $NHSO_5$  which, when diluted, separates into  $H_2SO_4 + HNO_2$ .



We believe that it is difficult to assume that there is an appreciable content of free  $HNO_2$  in acid with a higher density than 61° Bé, although an acid with normal characteristics contains  $NO_2$  which is free or present in a very unstable combination. The formation of  $HNO_3$  from  $NO_2$  seems probable.

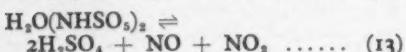
In any case, the low proportion of these components, which are more highly oxidised, side by side with a high proportion of very stable  $NHSO_5$ , cannot explain the high oxidising power of acids employed in our process where the nitrous content remains fairly low.

These acids have a composition which can vary between certain limits of density and nitrosity—61° to 65° Bé, 5 to 10%  $N_2O_5$ —with an  $H_2SO_4$  content which can vary from 73 to 76%.

In accordance with our hypotheses the most highly oxidising acids correspond to a general formula of:



and the separation of the nitro-sulphuric acid takes place in two stages:



which would explain for proportions of  $H_2O(NHSO_5)_2$  varying from 60 to 90% of the total nitrosity of the acid, on the one hand a high oxidising power in spite of a low nitrous content and, on the other hand the occurrence of  $NO_2$  in the acid.

### Some results

(a) **System of classical lead chambers.** S. A. Cros, La Coruna. 'Tour de tête' with partial filling and six rectangular chambers of a total volume of 3,528 cu. m.:

Production per cu. m./24 hr.: 11 kg. of  $H_2SO_4$ .

Consumption N.A.: 5 kg. of  $HNO_3$  per ton of  $H_2SO_4$ .

(b) **System of cylindrical chambers.** S. A. Cros, Porto-Pi. Five chambers of a total volume of 2,060 cu. m.:

Production per cu. m./24 hr.: 13 kg. of  $H_2SO_4$ .

Consumption N.A.: 5.8 kg. of  $HNO_3$  per ton of  $H_2SO_4$ .

(c) **System of conical chambers**

(**Mills-Packard**). Consorcio Agrario, Piacenza (Italy). Eight chambers of a total volume of 1,600 cu. m.:

Production per cu. m./24 hr.: 18 kg. of  $H_2SO_4$ .

Consumption N.A.: 6 kg. of  $HNO_3$  per ton of  $H_2SO_4$ .

(d) **Mixed system (classical lead chambers and ordinary steel cylinders).** Fabrica Perfosfati, Cerea (Italy). Three lead chambers, volume 1,500 cu. m., and two steel cylinders, volume 200 cu. m.; total 1,700 cu. m.:

Production per cu. m./24 hr.: 19 kg. of  $H_2SO_4$ .

Consumption N.A.: 6 kg. of  $HNO_3$  per ton of  $H_2SO_4$ .

(e) **Intensive system ('reactor' in ordinary steel).** Cie du Phospho Guano, La Rochelle-Pallice. Total volume of 'reactors', 625 cu. m.:

Production per cu. m./24 hr.: 85 kg. of  $H_2SO_4$ .

Consumption N.A.: 9 kg. of  $HNO_3$  per ton of  $H_2SO_4$ .

## Chemical Engineering for Students

The appearance of T. K. Ross's book\* at a time when Great Britain is still short of chemical engineering textbooks has naturally excited interest, although the object of the book, as shown by the title, is to introduce students to chemical engineering rather than to furnish them with a comprehensive text. Whilst the scope of the book is thus restricted, the responsibility placed upon the author is increased, since his is the responsibility of engendering the qualities of discernment and appreciation of the subject on which later learning can be built.

In seeking to make this introduction, Ross has given preliminary chapters dealing with process evaluation, materials handling and heat transfer, and has then proceeded to devote one chapter each to the more important unit operations. In form the book is therefore similar to textbooks of the subject, the difference being largely that of degree. The material presented is in general sound and the style clear. However, to encompass the subject within 200 pages, rigid selection has been necessary. Unfortunately, this process of selection has not made possible much practical illustration of the design methods presented or any review of their limitations and probable accuracy. Moreover, much that is important but difficult is not referred to, and the impression is given that chemical engineering involves no more than substitution in simple equations.

Three chapters in the book demand special mention. The opening chapter on process evaluation is excellent and deserves the attention of all chemical engineering students. The second, on materials handling, is misleading in giving no hint of

the immense field of fluid flow calculation with which the chemical engineer will have to deal. Finally, in dealing with distillation, the author has introduced a method of solving plate-to-plate column designs by constructions on the T-x chart for the system to be separated. The use of the intensive property temperature rather than the extensive property enthalpy leads to entirely erroneous results. It is hoped that this section will be rapidly revised.

G. G. HASELDEN

## The British Oil Industry

The growth of oil refining has been one of the most striking achievements of British industry, and it is significant that the first of a projected series of books called 'British Industries' deals with oil.\* It is written for the layman (or woman) interested in the present-day world, and for young people at the threshold of a career. In simple, non-technical language it tells the story of the growth and development of the oil industry. The book has been produced with the full co-operation of the Shell Petroleum Co. Ltd.; its authoritative nature is therefore unquestionable. However, the editors have made sure that the book covers the whole story of oil, not of one organisation, however vast.

After a chapter which shows how our lives are touched at every point by the products of the oil industry, there follows a step-by-step explanation of oil exploration, drilling, transportation, refining, distribution and marketing. There is also a short but enthusiastic chapter on petrochemicals. Finally, the social and economic effects of the oil industry are summarised, with particular emphasis on the industry's contributions to education.

\**An Introduction to Chemical Engineering*, by T. K. Ross. Pitman, London, 1953. Pp. x + 214 inc. index. 21s. net.

# Ore Dressing Research in India—1953

## Specially Contributed

The organised study of the principles and practice of ore dressing is a comparatively recent development in India. India hitherto was mainly an exporter of high-grade minerals and ores and no attention was given to the lower grades of economic minerals. During the last few years, owing to a progressive policy pursued by the Government, efforts have been made to upgrade domestic lean ores and also to find alternatives for materials in short supply. There are well-equipped ore-dressing sections at the National Metallurgical Laboratory, Jamshedpur, at the Indian Institute of Science, Bangalore, and at the Indian Atomic Energy Commission, Bombay, though there are individual operational units in other places in the country. Fundamental work on flotation and developmental work on low-grade ores have been conducted.

### Contact angle measurements

THE studies on contact angle measurements<sup>1</sup> at the pyrolusite surface are of considerable significance in view of the scanty literature on the subject. A high-grade granular crystalline pyrolusite (58.59% Mn) from South Tirodi Mines (Madhya Pradesh) has been used, and the modified captive bubble method of Cox and Wark<sup>2</sup> for measuring contact angle was employed in oleic, palmitic and stearic acid solutions. The contact angles obtained were 60°, 90° and 95°, respectively. It has been shown that the time of attainment of equilibrium contact angle varied from mineral to mineral and also with pH. In oleic acid at pH 8.2, the contact angle gradually increased to 60° at 1 hr. and remained constant for 24 hr.; at pH 5.9 it was 52° at 1 hr. and 58° after 1½ hr. In stearic acid the effect of pH was similar, but the contact angle after 6 hr. was 60° and remained so for 22 hr.; the equilibrium value of 95° was attained after 30 hr. Palmitic acid gave a final value of 90°.

The measurement of contact angle at higher temperatures consisted in heating the mineral with collector, cooling it gradually and measuring the angles at various temperatures. The precaution of heating the air to avoid its expansion in the cell has been taken and contact angles above 60°C. could not be measured. It has been found that heating has little influence in oleic acid solution, while with the other two collectors an increase in contact angle was noticeable as shown in Table 1.

An explanation has been proposed that the double bond and the carboxyl group form two centres of attraction in the oleic acid molecule while there is only one in the saturated fatty acids; the lowering of contact angle with increase in temperature is due to the greater ease of movement of the molecule with an increase in the energy of vibration of the molecule.

### Flotation of pyrolusite

The theoretical studies on contact angle measurements have been applied to the

Table 1.  
Effect of Temperatures on Contact Angle

Temp., °C.	Contact angle, degrees		
	Sodium stearate	Sodium palmitate	Sodium oleate
60	68	52	63
55	72	58	61
52	75	61	61
50	76	62	61
45	80	71	62
42	83	76	61
40	85	80	61
36	88	88	63
34	90	90	61
30	90	90	61
30 (without heat treatment)	38	48	61

Table 2. Effect of pH on the Flotation of Pyrolusite (Collector, oleic acid)

pH	Assay of concentrate		Recovery of man- ganese %	Assay of tailings	
	Man- ganese %	Silica %		Man- ganese %	Silica %
1.8	47.8	15.8	49.5	35.0	32.0
3.8	49.0	14.3	59.3	32.0	35.2
6.2	45.9	19.3	72.5	30.0	35.0
6.9	45.6	19.8	69.2	31.2	33.1
7.3	44.5	21.7	74.2	31.0	31.5
8.9	45.1	21.3	71.2	31.1	31.4
9.5	48.7	14.4	72.1	27.0	37.4
11.5	48.1	16.9	65.1	31.0	34.8
12.2	45.8	19.1	54.5	34.7	30.4

Table 3. Effect of Number of Carbon atoms in the Fatty Acid on the Flotation of Pyrolusite

Collector	No. of carbon atoms in the acid	Assay of concentrate		Re- covery of man- ganese %
		Man- ganese %	Silica %	
Caproic acid	6	45.3	20.0	49.1
Caprylic acid	8	45.3	21.5	76.2
Lauric acid	12	47.0	15.6	91.6
Palmitic acid	16	49.1	13.5	84.4
Stearic acid	18	51.1	11.8	78.0

beneficiation of a low-grade siliceous manganese ore (31.98% Mn, 47.1% SiO<sub>2</sub>) from Keshapur (Mysore).<sup>3</sup> Flotation characteristics have been studied under a variety of conditions, with oleic, stearic and palmitic acids as collectors; terpineol, cresylic acid, eucalyptus and pine oils as frothers; sodium silicate as depressant and at a wide range of pH and pulp density. Of the collectors, oleic acid was found to be the best, with 0.5 lb./ton giving good selectivity and a recovery of 77.5%. Sodium silicate with 0.6 lb./ton was the optimum addition; eucalyptus oil, 0.2 lb./ton at a pH of 9.5, gave best results. Using oleic acid (0.25 lb./ton), eucalyptus oil (0.2 lb./ton) and sodium silicate (0.6 lb./ton) the effect of pH has been studied (Table 2).

It is best to float the ore at pH 9.5, when selectivity and recovery are good.

The influence of the number of carbon atoms on the recovery of pyrolusite has been investigated under the above standardised conditions. While recovery increases with the number of carbon atoms to the maximum value in lauric acid, selectivity increases with an increase in the length of the fatty acid chain as shown in Table 3.

A comparison of the performance of the Leaf and Knoll cell,<sup>4</sup> which was modified by these authors with a stirrer and the standard Denver Sub A machine, shows that while the grade lies between 50 and 47% in the order, the recovery increases from 63.6% in the first through 72% in the second to the 80.9% in the last. Conditions have been standardised to get a concentrate of 53% Mn with a recovery of 88.5% in the Denver cell. The flotation of another sample, Amritapur (Mysore), has also been successfully completed.

### Heavy media separation

Heavy media separations are useful for the coarse particles. On the industrial scale the size of feed is 10 mesh and above, while on a laboratory scale the +65 mesh sizes can be used. With a view to obtaining information on the efficiency of heavy

media separation at the lower particle sizes, laboratory experiments have been conducted<sup>5</sup> using artificial mixtures of pyrolusite and silica in bromoform (sp. gr. 2.89) and saturated potassium mercuric iodide solution (sp. gr. 3.16). In the case of bromoform, complete separation is possible only up to 70 microns, below which the separation of silica from pyrolusite falls off and at a particle size of about 30 microns there is practically no separation. With the aqueous solution of potassium mercuric iodide, complete separation is possible only up to a size of about 50 microns. The separation of silica from pyrolusite improves on centrifuging. In the non-aqueous medium, the separation is complete up to about 30 microns and falls off till 5 microns, after which there is no separation. In the aqueous medium, separation is complete up to a size of 12 microns and even at 5 microns it is 80%. It is explained that the high efficiency of the aqueous solution in separation is perhaps due to the high penetrating power of the aqueous solution between the solid particles. These experiments have been well duplicated with a siliceous pyrolusite ore.<sup>6</sup>

#### Pyrites beneficiation

India relies on sulphur imports and consequently pyrites has received attention. The principal deposits are at Amjhor (Bihar), Chitaldrug (Mysore), Karwar (Bombay), Wynnaad (Madras), Rewa and Simla Hills. The Indian coals also contain some pyrites. The beneficiation of pyrites from Chitaldrug has been successfully undertaken.<sup>7</sup> The sample was associated with phyllite, chlorite, magnetite, quartz and calcite and assayed 34.69% S and 40.92% Fe. Jigging a 10/35 mesh fraction gave a concentrate assaying 43.99% S with a recovery of only 46.5%. Jigging followed by tabling the jig tail and the -35 mesh fraction produced a combined concentrate assaying 42.75% S with 75.1% recovery. Straight tabling a -35 mesh feed, after classification, resulted in a concentrate of 44.44% with a recovery of 66.3%. Tabling was followed by the flotation of the ground middling, tail and slime, and improved the overall recovery to 92.6% with a grade of 44% S. The ore is readily amenable to flotation after grinding 97% to -100 mesh and yields a good grade of concentrate with high recovery. In acid circuit, using 2.5 lb./ton of sulphuric acid, 0.51 lb./ton of copper sulphate, 0.15 lb./ton of potassium ethyl xanthate and 0.14 lb./ton of pine oil, the highest grade of concentrate assaying 47.96% S with 86.5% recovery was obtained. However, by mixing a middling product, a combined concentrate of 46.7% S with 93% recovery was obtained. In alkaline medium at pH 7.8 similar results were obtained.

The coals of Nowrozabad (Vindhya Pradesh) contain 0.8 to 1.3% pyrites.<sup>8</sup> Sink-float studies showed that about 80% of pyrites went with rejects at 1.58 specific

gravity. Economic recovery of the sulphide from the rejects by flotation was not possible, probably because the coals contain high proportions of fusain. Tabling of a product which sinks at specific gravity 1.6, crushed to 52 mesh, gave a concentrate with 45.64% S and 4.7% carbon with a poor recovery of 31.8%. On combining the middlings a combined concentrate with 40.9% S, 7.4% carbon with 61.56% recovery was obtained. It is estimated that, on the basis of current coal production in the area, about 42 tons of sulphur with less than 9% carbon can be recovered per day, and recovery is being contemplated by the coal mines.

#### Chrome ores

The beneficiation of low-grade chrome ores of Mysore has been completed.<sup>9</sup> A sample from Arsekere assaying 29.47% Cr<sub>2</sub>O<sub>3</sub> and 17.65% FeO had antigorite, chlorite, talc and anthophyllite as gangue. Screen analysis showed that chromite was fairly free from gangue at -65 mesh. Straight tabling of a classified -65 mesh feed yielded a concentrate assaying 47.45% Cr<sub>2</sub>O<sub>3</sub> with 74.3% recovery. Tabling of the ore ground to -100 mesh gave a concentrate of 48.25% Cr<sub>2</sub>O<sub>3</sub> and 64.7% recovery. On mixing a second concentrate the recovery could be pushed up to 75.6% with a grade of 47.97%. The iron content of the table concentrate could not be reduced by magnetic separation. A Humphrey's spiral test employing a -65 mesh feed resulted in a 44.2% concentrate with 64.8% recovery. Flotation of the ore ground to -200 mesh at a pH of 4.8, using sulphuric acid (3 lb./ton), sodium oleate (0.5 lb./ton) and oleic acid (1 lb./ton), gave a rougher of 48.35% Cr<sub>2</sub>O<sub>3</sub> and 61.9% recovery. However, a cleaner concentrate may increase the recovery to 70% with nearly the same grade.

Another low-grade chromite from Dodkatur,<sup>10</sup> containing serpentine, bastite, calcite, tremolite and quartz, gave the best concentrate of 40.34% Cr<sub>2</sub>O<sub>3</sub> with a recovery of 66.9%. Microscopic examination showed that there was no gangue, but the ore itself was a chrome spinel as confirmed by chemical analysis. It is thought that the product may be used in the manufacture of refractories.

#### Other work

In a work on the flotation of sulphur of microbial origin occurring in the clays of Masula, a controversy has arisen as to whether pine oil acts as a collector for the sample.<sup>11, 12</sup> India's only resources of lead and zinc are the Zawar mines. Experimental work has been completed<sup>13</sup> for the bulk and differential flotation of the sulphides under a wide variety of conditions. Studies have been concluded on the soap flotation of zircon, ilmenite, rutile and monazite and for separating them from the beach sands of Travencore.<sup>14</sup> Work is under way on the beneficiation of vermiculite deposits of Mysore.<sup>15</sup>

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#### SLURRY PUMPING AND PIPE LINE DESIGN

(Concluded from page 109)

##### READING LIST

G. E. Alves, D. F. Boucher and R. L. Pigford, 'Pipe-line Design,' *Chem. Eng. Prog.*, 48, (8), 385.

E. C. Bingham, 'Fluidity and Plasticity,' McGraw-Hill Book Co. Inc., New York, 1922.

H. E. Babbitt and D. H. Caldwell, 'Laminar Flow of Sludges, with particular reference to Sewage Sludge,' University of Illinois Technical Bulletin No. 12, Series 319.

*Idem*, 'Turbulent Flow of Sludges,' *Ibid.* No. 13, Series 323.

#### Recent publications

**Ethane from natural gas.** Basic data for the engineering design of plants to separate ethane, a major raw material for petrochemical production, from natural gas by low-temperature fractionation are presented in the American Institute of Gas Technology's Research Bulletin No. 22, 'Physical-Chemical Properties of Methane-Ethane Mixtures,' by O. T. Bloomer, D. C. Gami and J. D. Parent. The publication gives liquid-vapour phase equilibria, gas phase pressure-volume-temperature relationship, and saturated liquid and vapour density data for the methane-ethane system as determined by a detailed experimental investigation with specific mixtures of methane and ethane. The data are presented in tables and graphs.

**Handling slurry for steam-raising.** An illustrated pamphlet from Babcock & Wilcox Ltd. describes new developments in handling washery slurry where it is utilised for steam-raising in power stations and large industrial plants. Frequently met with in the form of a paste with the consistency of putty, slurry has hitherto presented greater problems in the handling rather than in actual combustion. It is generally recognised that the secret of handling it successfully is to keep it moving continuously. How this principle can be translated into practice is described in the pamphlet, with diagrams illustrating the salient features of the handling plant.



Anglesey. Bromine extraction plant. Sea-water distribution over blowing-out tower and connecting duct to absorber.

## ANTI-KNOCK COMPOUNDS

### Associated Ethyl's New Plants in Cheshire and North Wales

One of the most important steps in the development of ancillary facilities for the British petroleum refining industry is the recent establishment by Associated Ethyl Co. Ltd. of new plants for the manufacture of tetraethyl lead anti-knock compounds for gasoline. The new plants also constitute significant additions to the British chemical industry. The company manufactures its anti-knock compounds from materials as basic as sea water, from which bromine is extracted. This is done in a factory on the island of Anglesey, North Wales, which is equipped with its own  $SO_2$  and sulphuric acid units. The ethylene dibromide made at Anglesey goes to the main plant at Ellesmere Port, Cheshire, where the anti-knock compounds are made. At Ellesmere Port, T.E.L. is made from pig lead; other major process materials made there include sodium, ethyl chloride and ethylene dichloride. These interesting processes and plants are described in the following article.

ANTI-KNOCK compounds containing tetraethyl lead were first sold in the United States in 1924 by General Motors Chemical Corporation. After early troubles their use developed rapidly.

Ethyl Gasoline Corporation (now Ethyl Corporation) was formed in the U.S.A. to take over from General Motors Chemical Corporation the marketing of anti-knock compounds containing tetraethyl lead, under the brand name *Ethyl*.

The first commercial use of tetraethyl lead anti-knock compounds in countries other than the U.S.A. and Canada was in England in 1928. Serious difficulties similar to those experienced in the U.S.A. in 1924 were encountered and progress in the use of the material was extremely slow. In 1931, Ethyl Export Corporation was formed by Ethyl Gasoline Corporation to

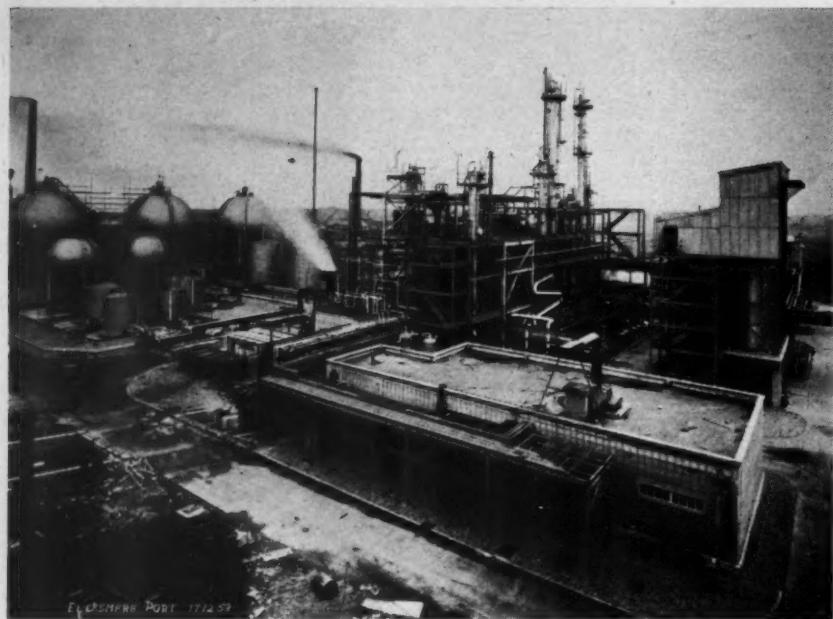
handle sales in countries outside the North American continent. The use of tetraethyl lead anti-knock compounds in these areas developed rapidly in the case of aviation gasoline and more slowly in the case of motor gasoline during the period 1931-39.

Plants for the manufacture of T.E.L. anti-knock compounds were built in Germany (1936), France (1938) and the United Kingdom (1940). Plants for the extraction of bromine from sea water were also built at the same time in France and the United Kingdom. Bromine is required for the manufacture of ethylene dibromide, which is an ingredient of the finished anti-knock compound. The German T.E.L. plant, which no longer exists in Germany, was built under licence by Ethyl GmbH. The French plant is owned by the French Government and leased to

the Société de Produits Chimiques 'Ethyl-Kuhlmann,' a company owned 50% by the Associated Ethyl Co. Ltd. and 50% by Etablissements Kuhlmann. The French bromine plant was built under licence from the Dow Chemical Co., U.S.A., and is owned and operated by 'Ethyl-Kuhlmann.'

In the U.K., both the T.E.L. plant and the bromine plant were owned by the Air Ministry and leased to British Ethyl Corporation, a company owned at that time 50% by Ethyl Export Corporation and 50% by Imperial Chemical Industries Ltd. Both plants were operated throughout the war by the Alkali Division of I.C.I. for and on behalf of B.E.C.

The Associated Ethyl Co. Ltd. was formed in September 1938 to take over the business and interests previously carried on by Ethyl Export Corporation.



Ellesmere Port. General view of chlorination group from west.

The new company (referred to hereafter as 'Associated') started trading on May 1, 1939.

The use of T.E.L. anti-knock compounds, both in aviation and motor gasoline, developed to such an extent during the war years that by the end of hostilities virtually all gasoline contained tetraethyl lead. In accordance with the agreement under which the plants were built, British Ethyl Corporation (now owned 100% by Associated) bought the British T.E.L. and bromine plants from the Air Ministry in 1945. Both plants continued to be operated by the Alkali Division of I.C.I. until December 31, 1947, when their operation was taken over by Associated.

#### The new U.K. projects

The capacity of the U.K. plants was insufficient to meet the demands of Associated's customers and accordingly the company arranged to purchase up to 66% of its requirements from U.S. producers for shipment to users all over the world.

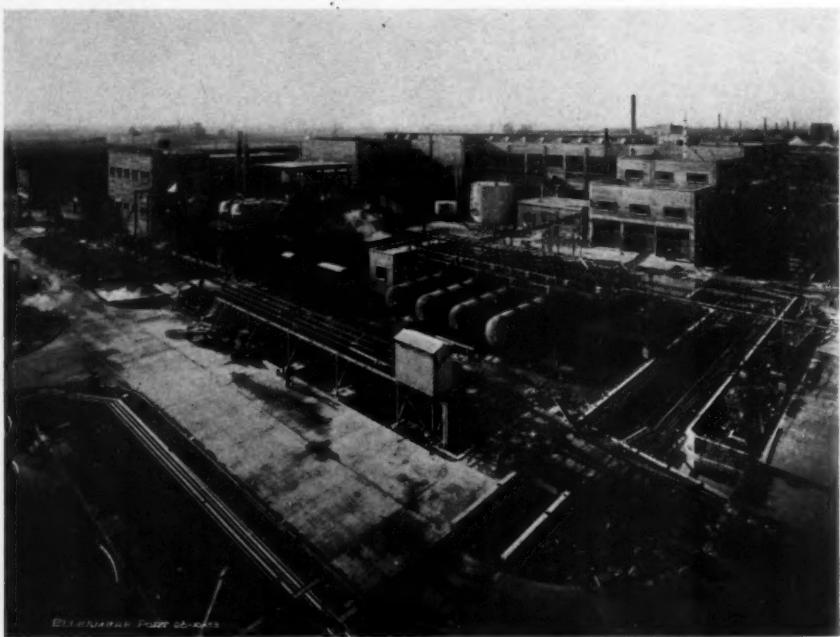
The logical centres for new manufacturing plants were the main refining areas, but surveys carried out in the Persian Gulf and the Caribbean revealed economic and practical difficulties.

In consequence, the decision was made in 1949 to build new manufacturing facilities in the United Kingdom. The decision to expand production involved, in addition to the building of new T.E.L. units, the construction of units for the production of essential process materials which were not available in sufficient quantities to meet indicated demand. The main materials concerned were sodium, ethyl chloride, ethylene dichloride and bromine.

After careful survey, a decision was

taken to build a new plant for the extraction of bromine from sea water on a 23-acre site at Amlwch, Anglesey. Construction started in April 1951 and bromine was first produced on November 11, 1953. The bromine obtained is converted into ethylene dibromide.

A site of approximately 60 acres at Ellesmere Port, Cheshire, was selected for construction of manufacturing facilities for T.E.L., sodium, ethyl chloride and ethylene dichloride. Work started on this site in February 1951.



Ellesmere Port. Sodium building and chlorine plant auxiliaries.

From May 1, 1954, Associated expects to be in a position to meet all demands made upon it for anti-knock compounds containing tetraethyl lead, from its own production in the United Kingdom.

Anti-knock compounds containing tetraethyl lead are marketed in France by Ethyl S.A., a 100% subsidiary of Associated. Ethyl S.A. draws its requirements from 'Ethyl Kuhlmann,' which as stated earlier, operates a T.E.L. manufacturing plant and a plant for the extraction of bromine from sea water.

As from May 1, 1954, all anti-knock compounds manufactured in the United Kingdom by Associated will be sold under the brand name *Octel*. This is merely to identify material manufactured in the U.K. by Associated, which will be identical to that previously sold by the company under the brand name *Ethyl*.

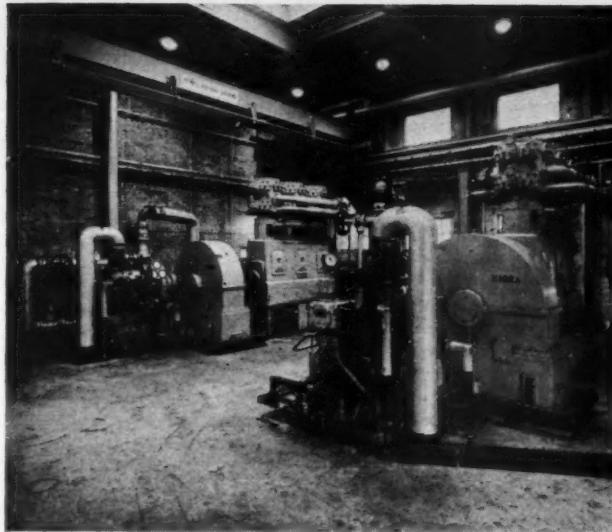
Anti-knock compounds manufactured by 'Ethyl-Kuhlmann' and marketed in France through the medium of Ethyl S.A. will continue to be sold under the brand name *Ethyl*.

#### ELLESMORE PORT FACILITIES

##### Lead-melting and -recovery plant

Pig lead, a major raw material used in the manufacture of tetraethyl lead, is principally of Australian origin. At Ellesmere Port the lead is melted in electrically heated pots and pumped in batches through electrically heated pipelines to the alloy plant.

Only about 25% of the lead charged to the autoclaves for the main reaction is converted to T.E.L. in each cycle. The remainder is collected as a lead sludge, dried, melted in a furnace and recycled to the melt pots.



Ellesmere Port. Left: The propane compressors, chlorination group. Right: The rectifier hall in the sodium plant.

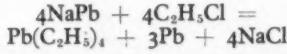
#### Lead/sodium alloy plant

In this plant, weighed quantities of molten lead and molten sodium from storage are mixed in a jacketed steel pot, to form an alloy containing about 10% of sodium. Heat is evolved during the formation of the alloy. Each 50-ton batch of alloy is tested for chemical composition, which must be exact before the molten alloy is fed to drum flakers where it is solidified and then filled into portable steel hoppers to a fixed weight.

All vessels containing sodium and alloy are nitrogen blanketed to prevent oxidation.

#### T.E.L. manufacture

Tetraethyl lead is produced in a batch process by the reaction of ethyl chloride with lead/sodium alloy according to the equation:



Batches of alloy are charged from the hoppers to jacketed autoclaves. Ethyl chloride is pumped, as required, from storage tanks in the T.E.L. area to a scale tank associated with each autoclave unit.

An excess quantity of ethyl chloride is slowly fed to the autoclave, and the reaction, in which heat is generated, is allowed to proceed to completion. Unreacted ethyl chloride, containing small quantities of hydrocarbon gases as impurities, is vented off to the ethyl chloride recovery and purification plants and is subsequently used again.

The reaction mass is discharged from the autoclaves into a distillation kettle containing water.

T.E.L. is recovered from the lead-salt residue by steam distillation. Batches of crude T.E.L. are washed, allowed to settle, and then filtered to remove suspended sludge. T.E.L. has a density of 1.65 and is easily separated from water. The

finished T.E.L. is collected in tanks for transfer to the blending plant.

The lead residues from the stills are discharged into a sludge pit which forms a part of the lead recovery process. A small quantity of T.E.L. is recovered from the steam-heated sludge-drying unit.

#### Blending plant

Ethylene dichloride, ethylene dibromide, kerosine and orange dye are blended with T.E.L. to produce tetraethyl lead anti-knock compound for use in motor fuel. An aviation blend of fluid, dyed blue, which does not contain ethylene dichloride, is also produced.

Weighed quantities of the materials are mixed in a vessel and, on test, each batch must meet a rigid specification for composition.

The batch is filtered and run to bulk stock tanks, or directly into 45-gal. steel drums for general distribution, or filled into rail or road tankers. All containers are of special design.

Facilities are provided for reconditioning drums and servicing tank cars.

A large proportion of the overseas drum traffic will be carried out *via* the wharf direct to vessels on the Manchester Ship Canal.

#### Electrolytic plant

The manufacture of the major process materials, sodium, ethyl chloride and ethylene dichloride, takes place at the Ellesmere Port works as follows:

Sodium and chlorine are manufactured by the electrolysis of a molten mixture of sodium chloride and calcium chloride in Downs cells. The process was developed by E. I. du Pont de Nemours & Co. and built partly under direct licence from them and partly under sub-licence from Imperial Chemical Industries Ltd.

A cell consists of a brick-lined steel

shell containing four vertical cylindrical graphite anodes, each surrounded by a steel cathode. A steel mesh diaphragm between the anode and cathode prevents the recombination of the products of electrolysis. High-purity salt, delivered in special hopper vehicles, is fed to the cells continuously by conveyors, and electric power is supplied as direct current from the rectifier plant.

Liquid sodium liberated from the cathode overflows into a receiver, from which it is transferred periodically to the filters where calcium and other impurities are separated. Purified liquid sodium is conveyed by electrically heated pipeline to the storage tanks at Ellesmere Port or loaded into tank cars for shipment to the Northwich works of Associated. All vessels are blanketed with nitrogen to prevent oxidation of the sodium.

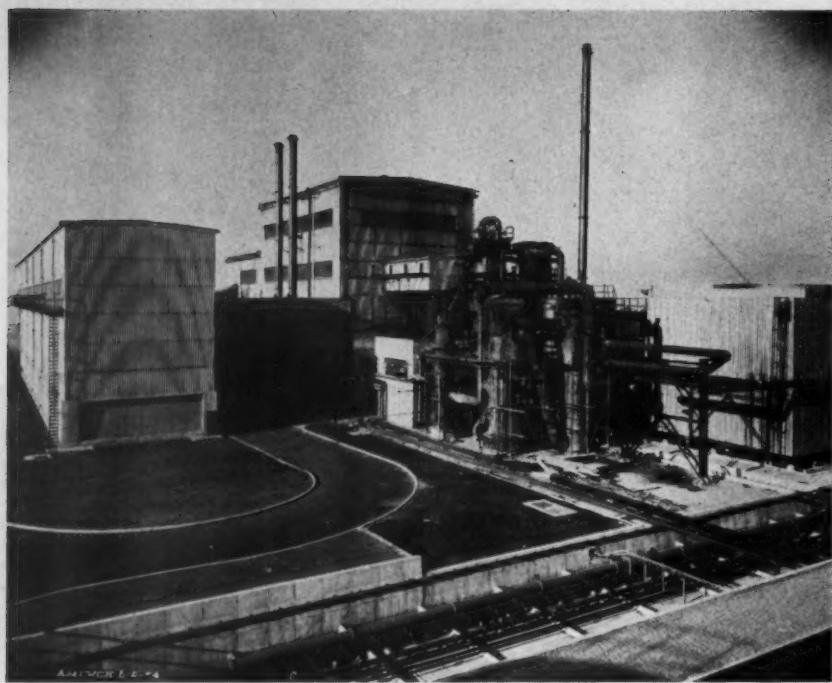
Gaseous chlorine, liberated simultaneously at the anode of the cell, is collected in a common main and directed to the chlorine liquefaction plant.

#### Chlorine liquefaction

Chlorine gas from the electrolytic plant is purified by washing with water and is subsequently dried by washing with sulphuric acid.

To facilitate storage and to free the chlorine from entrained air, the chlorine gas, after compression, is liquefied by means of a carbon dioxide refrigeration plant. The liquid chlorine is then transferred to stock tanks. The residual gas (tail gas) from the liquefaction process, which still contains some chlorine, is absorbed in a lime slurry which is prepared in a lime slaking plant.

Should the liquefaction plant become unserviceable, provision is made for absorbing the gaseous chlorine liberated from the cells in caustic soda liquor for an emergency period.



### Anglesey. The sulphuric acid plant and pyrites store.

Additional liquid chlorine is purchased from an outside source, received in road or rail tankers and put into stock.

Liquid chlorine is transferred from the stock tanks to the chlorination group of plants, where it is converted to gaseous chlorine in an evaporator before being used.

## Ethyl chloride

Ethyl chloride is manufactured from chlorine, ethane and ethylene.

In the first part of the process, chlorine reacts with ethane to form crude ethyl chloride and hydrochloric acid gas. These are separated by distillation and the hydrochloric gas then reacts with ethylene to form more crude ethyl chloride.

The crude ethyl chloride is purified by distillation and the almost pure ethyl chloride obtained is collected in a spherical storage tank for transfer by pipeline to the T.E.L. plant, where it is used in the main reaction.

Some of the product is conveyed by road tanker to the T.E.L. plant of the company at Northwich.

## Ethylene dichloride

Ethylene dichloride, which is blended with T.E.L. and ethylene dibromide in the motor grade of anti-knock fluid, is produced by the reaction between gaseous chlorine and ethylene in a circulating stream of ethylene dichloride.

The crude product is washed with water, separated and finally distilled. The almost-pure ethylene dichloride is collected for transfer by pipeline or road tank car to the T.E.L. blending plants.

#### **Heavy ends from the distillation units**

Electricity Authority, constructed adjacent to the works, is provided with multiple 132-kv. feeders. Electricity is taken from the local Electricity Board at 33 kv., reduced to 11 kv. for distribution to substations and to the rectifier plant from which direct current is supplied to the electrolytic plant.

Nitrogen is used to blanket all vessels containing sodium or lead/sodium alloy, and used generally as an inert gas. It is made by burning hydrogen and air to remove the oxygen. It is then scrubbed to remove carbon dioxide, dried and compressed, and delivered to storage tanks in the consuming areas. Hydrogen will be supplied by pipeline from Stanlow refinery and, should this supply not be available, hydrogen will be produced from anhydrous ammonia in I.C.I. standard cracking units.

Gas is taken from the town supply, compressed, and distributed to user plants at 10 in. to 20 in. w.g.

## ANGLESEY FACILITIES

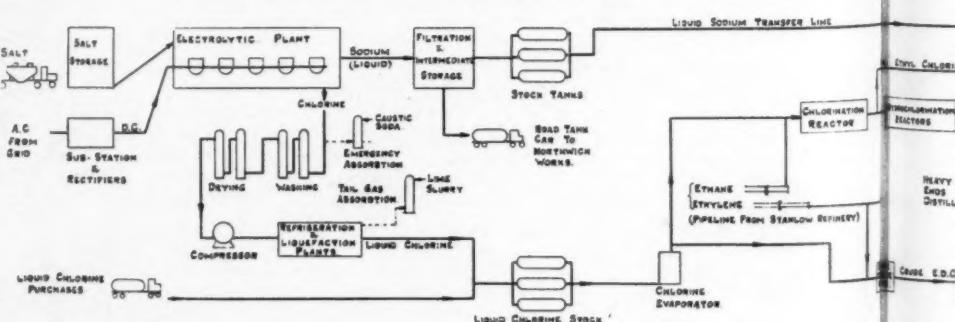
The Anglesey plant of Associated for the extraction of bromine from sea water, and the production of ethylene dibromide, has in addition a sulphur dioxide and sulphuric acid plant, which are described in more detail later in this article.

Since sea water contains only about 67 p.p.m. of bromine, enormous quantities are required for the process. A sea-water intake and tunnel, situated well below the minimum low-water level, provide a constant supply to the pumphouse. The tunnel is several hundred feet long and has been constructed through solid rock. Two pumps, each having a capacity of 30,000 gal./min., and the third pump having a capacity of 50,000 gal./min., raise the sea water to a reservoir from where it flows to the stripping or blowing-out tower.

### Bromine extraction

As the sea water flows to the blowing-out tower, dilute acid is added to reduce the pH to 3.5, thus preventing hydrolysis during the liberation of bromine. Chlorine gas, purchased as liquid chlorine in rail tankers and evaporated by steam heating, is introduced in sufficient quantity to

## FLOW SHEET FOR MANUFACTURE OF TETRAETHYL LEAD I-I-KNO



release the bromine present in the sea water as sodium bromide.

In the blowing-out tower the sea water, flowing in a thin film over millions of rings, is stripped of about 80% of the liberated bromine by a large upward current of air produced by fans situated at the top of the absorber.

The effluent sea water flows from the bottom of the tower back into the sea at a safe distance from the intake.

As the bromine-laden air passes along a duct to the absorber, it is treated with sulphur dioxide and water, and a fine mist of hydrobromic and sulphuric acids is produced. These acid mists are extracted from the air by means of water-irrigated glass fibre filters situated in the absorber.

The resulting solution, known as primary acid liquor (P.A.L.) or absorber liquor, contains about 10% of bromine as hydrobromic acid, as well as sulphuric acid and small quantities of hydrochloric acid.

#### Bromine recovery

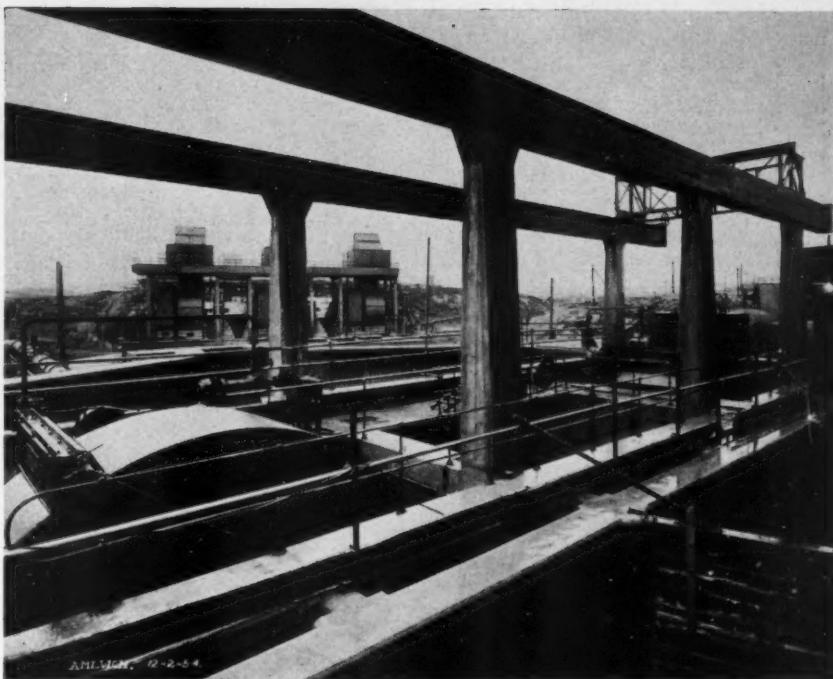
The P.A.L. is first treated with chlorine to liberate bromine from the hydrobromic acid, followed by steam distillation of the liberated bromine. The whole of this process is carried out in the steaming-out towers. The gaseous bromine produced at the top of these towers passes through condensers, separators and driers, and is ultimately collected as liquid bromine.

The effluent from the steaming-out towers contains sulphuric acid originally present in the primary acid liquor, as well as hydrochloric acid produced from the chlorine used in displacing the bromine. This effluent is returned to the dilute acid tank for acidifying fresh supplies of sea water to the blowing-out tower.

The bulk of the liquid bromine is used in the manufacture of ethylene dibromide, and small quantities are packed into bottles and drums for sale as liquid bromine.

#### Ethylene plant

Purchased ethylene is delivered as a liquid in special road tankers and is transferred to liquid stock tanks. From here it is piped to an indirectly steam-heated evaporator and the ethylene gas produced is compressed and stored for use as required.



Anglesey. The sea-water pumphouse and the rotary screens.

Should road conditions or other reasons hold up the supply of liquid ethylene, a standby plant for making ethylene gas from ethanol can be put into operation. In this, ethanol vapour is heated in the presence of a catalyst and ethylene gas and water vapour are produced. The ethylene is purified, compressed and stored.

#### Ethylene dibromide

Ethylene dibromide is made in a batch process by bubbling ethylene gas into liquid bromine contained in special reactors. Large quantities of heat are given off during the reaction, and extensive water-cooling systems have to be employed, so that the rate of reaction can be controlled and the losses of both bromine and ethylene kept to a minimum.

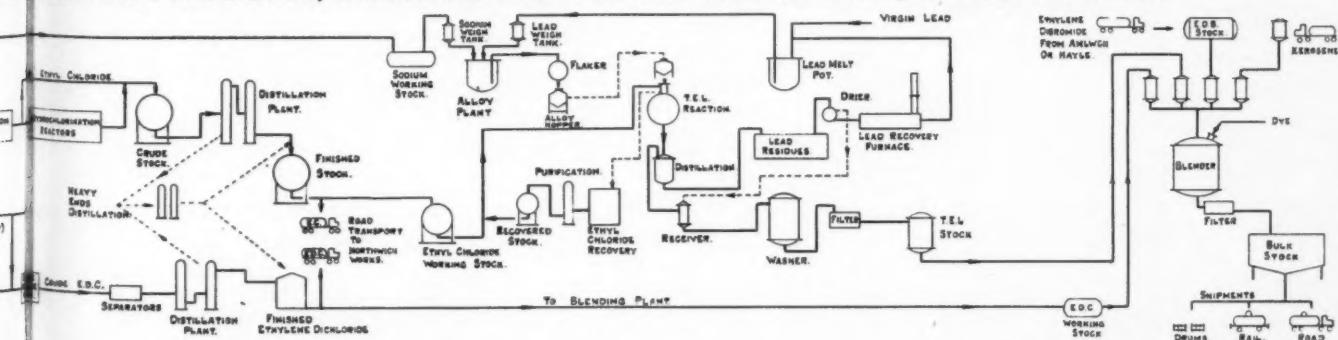
The crude ethylene dibromide generally contains small quantities of acid (mainly hydrobromic acid) which are produced owing to impurities in the reactants.

These acids are removed by mixing the ethylene dibromide with sodium carbonate solution in a neutraliser. When neutralisation is complete, the ethylene dibromide is separated by taking advantage of its high density, and is then passed to storage tanks for loading into road or rail tankers for shipment to the Ellesmere Port or Northwich works of the company. The soda ash solution, when ultimately nearly all converted to sodium bromide, is introduced to the steaming-out tower for the recovery of the bromine content.

#### Sulphur dioxide and sulphuric acid

Sulphur dioxide is made by burning iron pyrites ( $FeS_2$ ) in the presence of air in a flash roaster. The sulphur in the pyrites forms sulphur dioxide and a residue of iron oxide is left. Imported pyrites are received at the works by rail. The sulphur dioxide is purified by passage through precipitators, washers and driers.

### ADMIT-KNOCK COMPOUNDS, INCLUDING ESSENTIAL PROCESS MATERIALS, AT ELLESMERE PORT



and is mixed with air to a concentration of not more than 7% sulphur dioxide.

Part of this sulphur dioxide air mixture is led direct to the duct joining the blowing-out tower to the absorber in the bromine extraction plant. The remainder of the sulphur dioxide is used in the manufacture of sulphuric acid in a standard design of contact plant.

#### Services

A waste-heat boiler attached to the sulphuric acid plant produces an appreciable quantity of the steam required in the bromine recovery process. Additional quantities of steam are produced as required in oil-fired vertical boilers.

Service pumps in the sea-water pump-

house provide sea water for cooling purposes in the sulphuric acid plant, for bromine reactors, and for the ethylene standby plant.

Electricity is taken from the local Electricity Board 33-kv. grid and is reduced to 11 kv. for distribution to substations. A 3.3-kv. supply is provided for the high-voltage motors on the sea-water pumps and the absorber fans.

Compressors supply air for use in the chlorine evaporation plant in the sulphuric acid plant, for instruments and for general purposes.

The vacuum service is provided by means of an eductor-type vacuum pump for bromine removal in the packing area and for syphon operation in the bromine process.

cerned with the different types of catalyst which may be employed and the effects of minor variations on their activity. The importance of crystal structure in the unreduced catalysts is very apparent and the utility of x-ray diffraction methods for this type of study is well illustrated. From this review, it is clear that there is still much to be learned concerning the catalytic synthesis of ammonia, in spite of the fact that it has now been operated on a commercial scale for about 40 years and that the annual production of ammonia in the U.S.A. alone has reached the figure of about 2 million tons.

There are two sections, 'Surface Studies with the Vacuum Micro-balance' and 'Instrumentation and Low Temperature Applications,' by Rhodin, which give an account of the design and manipulation of this interesting piece of equipment and review of the type of problems to which it can be applied, notably the effect of crystal form and orientation and on the adsorption of nitrogen by metals at low temperatures, and the study of the low-temperature oxidation of metal surfaces. The corresponding article, 'High Temperature Reactions,' by Gulbransen, describes apparatus suitable for operation at high temperatures and its application to the study of the oxidation of metals, the vapour pressure of metals, and the effect of oxide and nitride films, and the study of the combustion of solid fuels.

'The Heterogeneous Oxidation of Carbon Monoxide' by Katz gives a review of the catalysts suitable for the oxidation of carbon monoxide at low temperatures, notably in respirators. There is also a consideration of adsorption wave kinetics, which treats on the distribution of gas throughout a solid reaction bed during the continuous passage of gas-laden air.

'The Elucidation of Reaction Mechanisms by the Method of Intermediates in Quasi-Stationary Concentrations' by Christiansen discusses the application of chemical kinetic studies to the elucidation of reaction mechanisms. The study of reaction mechanisms is still a fruitful field of research investigation, and it is sobering to realise the relatively small number of organic reactions, the mechanism of which is soundly established. The author perhaps gives the impression that a little more recognition of kinetic studies would result in the elucidation of the mechanism of most organic reactions. However, it would seem that in many cases the limit is not set by the desire to carry out kinetic studies, but more by the insufficient development of the analytical methods which are necessary to enable precise measurements to be made.

P. J. GARNER, B.A., PH.D., F.INST.PET.

## Advances in Catalysis

IN this volume,\* the editors have maintained the high standard previously set in this series and present information on subjects ranging from technical catalytic processes to the elucidation of reaction mechanisms and specialised techniques.

The section by Tolpin, John and Field on 'Contributions of Russian Scientists to Catalysis' would probably be sufficient to justify buying this book. The Russian chemical literature is not easily accessible and this review, which includes 460 references, gives a very wide cover of developments up to the present time. It is clear that very considerable attention is being paid by Russian chemists and physicists to catalytic phenomena, and the high quality of much of their work is brought out. The aspects covered are schools of thought on catalysis, investigation of adsorption phenomena, kinetics of heterogeneous catalytic reactions, modification of catalysts and catalytic conversions.

'The Uses of Raney Nickel' by Lieber and Morritt gives a brief review of the preparation, properties and uses of Raney metal catalysts. Most of the information available is concerned with Raney nickel catalysts, but other metals, such as cobalt, are covered and the effect of additives is also considered. The interesting point, that the effectiveness of these catalysts is quite largely due to the presence of aluminium, is well brought out. About half the chapter is devoted to special reactions such as hydrogenation, which may be performed in the presence of Raney catalysts.

From the first volume, attention has been given to the Fischer-Tropsch process, and Volume IV contained a historical review by Pichler of 25 years of the Fischer-Tropsch process. Until recently, it seemed that the process was about to become established on a commercial basis in the U.S.A., but recent reports indicate that

the plant has run into very serious difficulties, so that the large-scale development of Fischer-Tropsch synthesis outside of wartime Germany has still to be awaited.

'Iron Nitrides as Fischer-Tropsch Catalysts' by Anderson gives a review of the possibilities of a particularly suitable type of catalyst developed by the Synthetic Fuels Branch of the U.S. Bureau of Mines. These iron catalysts are prepared by passing ammonia over reduced iron and show themselves to be remarkably resistant to oxidation and to the decomposition of elemental carbon, both processes which can lead to the rapid deterioration of Fischer-Tropsch catalyst. The author concludes that iron nitrides show good prospects for use in commercial reactors.

### Hydrogenation with synthesis gas

A subject related to Fischer-Tropsch synthesis is 'Hydrogenation of Organic Compounds with Synthesis Gas' by Orchin. One of the most intriguing features of catalytic hydrogenation is the effect of poisons, and carbon monoxide has come to be accepted as having an adverse effect on most hydrogenations with metallic catalysts. However, study of the oxo reaction, by which an olefin is converted into an aldehyde by reaction with carbon monoxide and hydrogen in the presence of a cobalt catalyst, shows that hydrogenation of the olefin can play a significant part. This particular aspect is specially reviewed and figures are given for the hydrogenation of different types of unsaturated compounds under various conditions. An interesting reaction is the so-called 'homologation reaction,' in which an alcohol reacts with carbon monoxide and hydrogen to give the next higher alcohol containing one more carbon atom than the starting material. Experiments with methanol and with benzyl alcohol show that the reaction does not proceed via the intermediate formation from the starting alcohol.

'Latest Developments in Ammonia Synthesis' by Nielsen is primarily con-

\**Advances in Catalysis*, Vol. 5, edited by Frankenburg, Komarewsky and Rideal. Academic Press, 1953. Pp. 487. \$11 net.

### To Authors of Books

The Editors and Publishers of the Leonard Hill Technical Group are always ready to consider technical and scientific manuscripts with a view to publication.

# Wastage of Refractories by Slag Attack

## Causes and Effects

By D. Dixon

*Slag attack and spalling are the chief causes of wear and wastage of refractories in service. Here the author discusses the causes and effects of the various forms of attack which, he points out, may occur simultaneously.*

*He is concerned principally with aluminous fireclay or high alumina refractories.*

REFRACTORIES are essential components of a great variety of furnaces. The processes on which the furnaces are employed can be classified generally as smelting, melting and heat treatment, but the furnaces are a multitude of sizes and types. So there is also a great variety of temperature and other service conditions for the refractories.

Many tests have been devised to determine the chemical and physical characteristics of the different grades of refractories which are available so that their suitability for a type of furnace can be assessed. These laboratory tests include chemical analysis, spectrum analysis and differential thermal analysis to determine the chief constituents of a refractory and the impurities which invariably are present. A simple refractory test indicates the temperature which a refractory will withstand without change. The more elaborate test of refractoriness-under-load indicates what temperature and load can be applied without deformation. Then there are the tests to determine the extent of after-contraction or after-expansion on reheating and the reversible thermal expansion. Other tests indicate apparent and true porosity resulting from the volume of open and closed pores. Then there are the cold crushing strength, the cross bending strength or modulus of rupture, the density, the thermal conductivity, the thermal spalling resistance and the resistance to slag attack which are indicated by various tests.

The correlation of laboratory tests with service conditions, however, is still a problem and particularly so in respect of resistance to slag attack. The measurement by laboratory test of the resistance of a refractory to attack by slags or fluxing gases in a variety of furnaces is obviously a difficult job. Certainly there are approximate or comparative methods such as the heating of a measured quantity of slag in contact with the refractory sample and the observation of the extent of reaction or the depth of penetration. Another method is to heat the refractory sample by an impinging flame carrying fluxing particles or to direct a stream of slag particles to a heated refractory surface.

The result of tests such as the foregoing may be helpful in the choice of a grade of



**Forms of wastage of refractories described in this article:** (a) Cracking and shrinkage in service as a result of under-kilning or high service temperature; (b) solution and erosion by fluid slag; (c) thermal spalling from rapid heating; (d) thermal spalling from rapid heating and also rapid cooling; (e) softening and deformation by high temperature. The effects shown in (a) and (e) may have been assisted by reducing conditions or by penetration of the texture of the refractory by fluxing gases.

refractory to resist slag attack, but it is not possible to correlate such simple tests accurately with conditions in the furnace. Indeed, it may be that the user cannot state just what the conditions of temperature and atmosphere in the furnace will be.

Yet the chief causes of wear and wastage of refractories in service are slag attack and spalling rather than high temperature. Slag attack and spalling may be combined to accelerate wastage. Softening and after-contraction or after-expansion by temperature effect have to be kept in mind as dangers, but as a cause of refractory consumption and costs they are relatively unimportant. The purpose of these notes, therefore, is to describe slag attack and spalling in some detail in order to assist a better understanding of them.

### Mechanism of wastage

It may be useful to point out that the mechanism of wastage by slag attack is not 'burning' as is often stated. Refrac-

tories do not burn unless they contain carbon and the conditions are strongly oxidising. Under oxidising conditions, of course, carbon bricks and the carbon in plumbago crucibles may burn. Aluminous, siliceous and basic refractories, however, are not burned in service, but are consumed by corrosion, erosion, penetration, solution or a combination of these processes. Fluxes from the fuel, the charge in the furnace or the fuel gases are the cause. Contributing to these forms of wastage we may find thermal spalling from temperature change and gradient, physical spalling from surface fusion or mechanical spalling from structural strains.

The accompanying sketches are an attempt to illustrate the forms of slag attack and spalling. In considering the sketches, however, we have to keep in mind that in practice the mechanism of slag attack is complex and wastage of refractories in a furnace may be caused simultaneously by more than one of the processes illustrated.

## DIFFERENT FORMS OF SLAG ATTACK

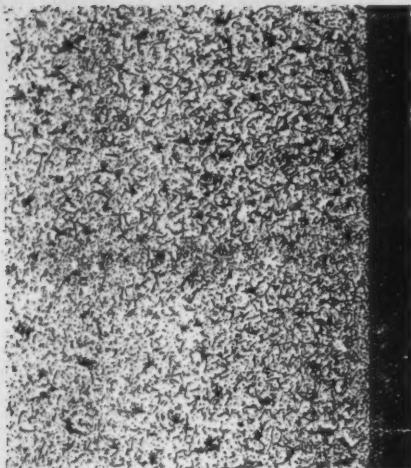


Fig. 1. Thick layer of viscous slag on working face of refractory.

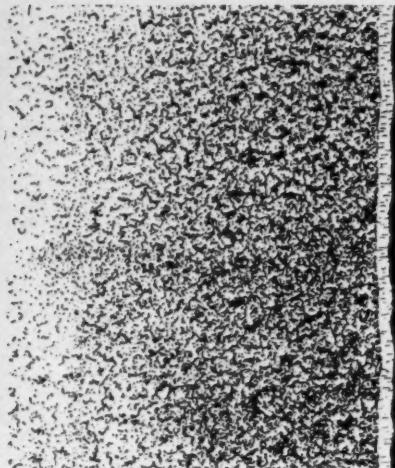


Fig. 2. Coating of fluid slag which can cause rapid wastage.

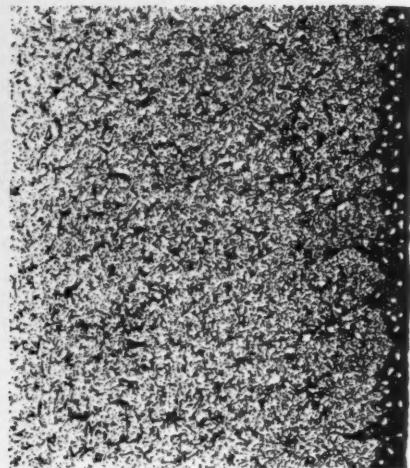


Fig. 3. Penetration of slag into the working face of refractory.

Fig. 1 shows by the thick black layer a coating of slag deposited on the working face of a refractory brick or shape. The slag is viscous and it has reacted but slightly with the refractory surface, but it adheres to it. There is no visible reaction product of slag and refractory as a refractory-slag interface. The slag coating is not an active solvent of the refractory and it is protecting the refractory from abrasion and erosion. It may stay for a long time with little change, provided that cooling and reheating of the furnace does not cause it to spall or break away, when it may bring a layer of the refractory with it.

Fig. 2 is more interesting as it illustrates a form of wastage of the refractory which may be rapid. The slag coating is more fluid than in Fig. 1. The cause of greater fluidity is a higher content of constituents such as iron, lime and alkalis in compounds of low melting point or alternatively a higher furnace temperature. The refractory is an aluminous fireclay grade. The slag has reacted with the refractory at the furnace operating temperature so that some of the silica in the fireclay mix has been dissolved into the flowing coat of slag. The alumina in the fireclay mix, with some of the silica, has been formed into a layer or refractory-slag interface of mullite crystals which have an alumina content of approximately 72%.

This interlacing network of mullite crystals known as a 'white line' forms a protective layer on the working face of the refractory, for mullite is very resistant to chemical attack and erosion by fuel slags. It delays the penetration and solution of the refractory by the fluid slag. As the alumina in the mullite is slowly dissolved, it increases the viscosity of the slag deposit and renders it less active and erosive. The higher the content of alumina in the refractory, the more readily under appropriate furnace conditions will

the resistant mullite hide be formed. We see from the foregoing one of the reasons for increasing the alumina content of clay-bonded refractories.

However, it may be that the furnace temperature is too low, the slag is too erosive, the refractory contains too little of alumina or the refractory is too permeable, for the mullite layer to be developed. Then we may see penetration and solution as illustrated by Fig. 3. A resistant layer has not been formed, the bonding clay in the refractory is being dissolved and the more resistant grog particles are being carried away in the flowing slag. Most refractory mixtures consist of prefired clay or other non-plastic grog, such as sillimanite, fused alumina or silicon carbide, ground to specified meshes and bonded with plastic clay which forms a glassy matrix when the refractory is kilned. Fig. 3 suggests that a wash or coating of fine-meshed aluminous air-setting cement on the working face of the refractory might assist the primary formation of a resistant hide. Such cement coatings have had a limited value on coarse-textured refractories which are needed sometimes to withstand without fracture the rapid temperature changes in some intermittent furnaces.

A contrast with the foregoing is the formation of a thick, partly vitrified refractory-slag layer as shown in Fig. 4. The thick refractory-slag layer has been formed as a result of penetration of the texture of the refractory by fluxing furnace gases combined with the softening effect of high temperature. When there is alternate heating and cooling as in intermittently operated furnaces, the thick semi-vitrified layer may break away because of permanent or reversible volume changes greater or less than those of the refractory. This form of wastage is known as physical spalling.

When we consider physical spalling,

however, we have to remember the other forms of spalling, namely the thermal spalling from temperature shock and the mechanical spalling from structural strain, with which it is often associated.

Refractories, like almost all other materials, expand on heating and contract again on cooling. This is known as reversible thermal expansion. It is distinct from the permanent change in volume, known as after-contraction or after-expansion, from kilned dimensions, which refractories may show on test or in service. The reversible thermal expansion of well-kilned aluminous fireclay refractories is an even rise of the order of 0.5% per 1,000°C. The strains arising from reversible thermal expansion will be greater in large shapes or complicated shapes than in small simple shapes.

It is clear that strains and the tendency to fracture will arise most strongly when temperature changes are rapid and extreme, or when there is a steep temperature gradient from the hot face of the refractory because of external cooling. The strains arising from sharp temperature change or steep temperature gradient will be most severe in the medium range of temperatures. In the highest range of furnace temperatures, the volume changes arising from temperature variation are accommodated by slight yielding of the glassy matrix or bond in the refractory.

Fig. 5 illustrates how rapid heating and expansion of the working face of a refractory block may cause a cleavage parallel to the hot face so that a thickness flakes away. This is simple thermal spalling. It may be combined with or aggravated by physical spalling as shown in Fig. 4.

On the other hand, Fig. 6 illustrates how rapid cooling of the hot face of a refractory block puts this hot face into tension so that spalling cracks tend to be at right-angles to the hot face.

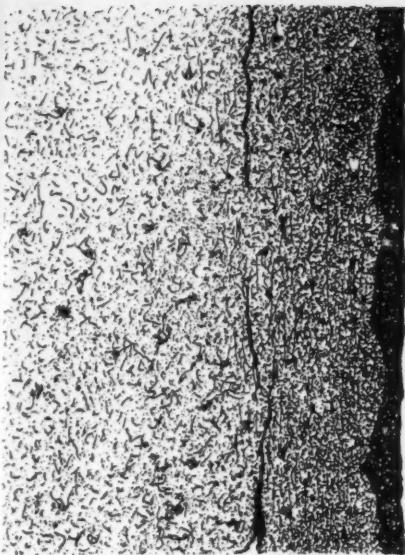


Fig. 4. Thick, partly vitrified refractory-slag layer.

These lines of cleavage may, of course, be modified in practice by lack of homogeneity of texture of the refractory or by shape which influences the temperature gradient. Thermal spalling may also be aggravated by mechanical strains arising from movement of the furnace structure. The line of fracture in cases of spalling, however, can indicate whether rapid heating or rapid cooling is the cause.

When considering slag attack, it may be useful to regard corrosion as a term describing the formation of a reaction product from slag and refractory in the form of a covering, and erosion as a term describing solution and removal of the refractory surface by a moving coat of slag which is continuously renewed.

The physical and chemical changes occurring in slag attack are complex and research workers are faced with difficult jobs. An example is the determination of the melting points of compounds formed in the slag coating on refractory linings in different types of furnace. The reaction product of slag and refractory is usually of lower melting point than either of the contributing materials. We have seen that when the viscosity of the reaction product is low the rate of corrosion and erosion is much accelerated. As rate of diffusion is relative to viscosity which is relative to temperature, a slight increase in temperature may accelerate slag attack to a surprising degree. The diffusion of gases in the reaction product of slag and refractory is an important influence. Reducing conditions in the furnace and the formation of ferrous oxide in the refractory from ferric will result in carbon deposition in the pores of the refractory and some disintegration of the texture.

Silicon carbide refractories have two special characteristics which assist resistance to slag attack. One is the 'non-

wetting' characteristic which checks adhesion of the slag. The other is the high thermal conductivity which results in some cooling of slag in contact so that it is more viscous and less erosive. Silicon carbide refractories are very useful, therefore, for slag resistance when furnace atmosphere is neutral or reducing rather than strongly oxidising. Under strongly oxidising conditions, slow oxidation of the silicon carbide may occur with formation of silica and disintegration. Carbon refractories are very inert to slags, but conditions of use must not, of course, be oxidising.

These notes relate primarily to aluminous fireclay or high-alumina refractories. The strongly acid silica refractories and the strongly basic magnesite refractories have special characteristics which influence attack by fuel and metal slags. The number of grades of the so-called neutral aluminous refractories seems continually to increase with alumina contents ranging from 40 to 90% and more. We have to remember, however, that there is a great variety of use for them and there is an increasing need for specialised grades.

The simple analysis and the degree of refractoriness have for long been regarded as indications of quality and suitability. We see now, however, a growing tendency for high kilning temperature to be specified or implied as an indication of chemical and physical stability, with apparent porosity or permeability as an indication of resistance to penetration by slags and fluxing gases.

So what is the sum total of the foregoing notes and what is their purpose? Can we look forward to specifications or test data which in respect of slag resistance will be a reliable guide to users of refractories?

It is difficult to answer in the affirmative. Furnace conditions cannot be closely assessed in many cases and laboratory-test conditions cannot be correlated closely with them. The manufacture of refractories is based upon arrested rather than completed chemical reactions and the

long-term effects of time, temperature and atmosphere in service are difficult to match or estimate. Progress will continue to depend upon co-operation between scientist, manufacturer and user, with understanding by the user of the difficulties involved.

Meantime, the characteristics and qualities of any high-grade refractory appear to be: (1) The analysis; (2) the approximate kilning temperature; (3) the fusion point; and (4) the porosity or permeability.

### Rheology of Non-aqueous Suspensions

Knowledge of the flow properties of mixtures of powdered solids and liquids other than water is of importance in many industries, and investigations made at the Road Research Laboratory over a number of years on suspensions of this type are described in Road Research Technical Paper No. 28.\*

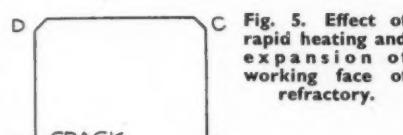
The powders used for the work included limestone, granites, slate, silica, slag and Portland cement. Some of the tests were made with very fine powders collected from air-separating systems or were specially prepared in a ball mill. Two types of liquid were used for the suspension of the powders. One was a refined coal tar obtained from the high-temperature carbonisation of coal and consisting of hydrocarbons of a predominantly aromatic character, the other a bitumen containing hydrocarbons of a predominantly aliphatic nature which was obtained as a residue in the distillation of crude petroleum. Both liquids had a similar viscosity at a temperature of 15°C. and behaved substantially as simple Newtonian liquids at temperatures above that figure.

The main problem was to find the effect on the flow properties of the mixture of the amount and physical characteristics of the powder used, particularly when it was present in large quantities. The work was therefore concerned first with the study of the flow properties of two-phase systems and secondly with the physical characteristics of finely divided materials.

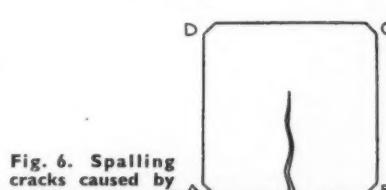
The first part of the paper describes a systematic study of the flow properties of the powder/liquid suspensions over a wide range of concentration. The results showed the importance of the voids fraction in the dry powders. The second part describes work on the void structure of the packed powders and the third part relates the results to the rheological behaviour of systems of high solid concentration.

The paper will be of interest to specialists in this field in the road and other industries concerned with the use of fine mineral powders.

\**The Rheology of Non-aqueous Suspensions.* H.M.S.O., London. Pp. 50. 2s. (50 cents, U.S.A.).



SUDDENLY HEATED.



SUDDENLY COOLED.

# Chemical Engineering Studies and Process Development\*

By H. Hoog

THE tremendous increase in the production of organic chemicals in the last decade has been brought about with the assistance of research activities both in the discovery of new methods and in the development of those methods.

When an idea has been brought to fruition in the research laboratory the function of process development can be summarised under four headings:

- (1) To test the correctness of the principle of the process.
- (2) To choose between alternative procedures.
- (3) To evaluate the economics of the process.
- (4) To provide data for the design of a commercial plant.

Laboratory research is normally followed by the drawing of a diagram for the new process. It is then essential that the stages in this diagram be analysed and separate investigations made for the most favourable conditions at each stage. Only when this has been done can a pilot plant be usefully built.

A large organisation needs to have part of its development section devoted to the study of unit operations. This knowledge is always being required for the development of any particular process which is being tackled by a further team of workers in the section. By considering three examples in the following paragraphs, it will be seen that this study of unit operations is an important part of process development.

## Sulphation of olefines

This reaction is carried out on a large scale in the production of liquid synthetic detergents. It is not intended to discuss the whole process, but rather to consider the actual reaction system. To make *alpha* olefines and sulphuric acid react with each other it is necessary to mix intimately the two non-miscible phases. In addition, it is necessary to remove from the system the heat of reaction. These requirements can be met in several ways:

- (a) By passing the two components through an annular space with cooling of both inner and outer wall, using a rotor for stirring.
- (b) Using an annular, cooled reaction chamber (as before), the feed being pre-mixed and made to flow through at high velocity.
- (c) The two components can be passed through a mixer, the heat being removed by a cooling coil or jacket.
- (d) A centrifugal pump can be used to circulate the mixture through a cooler.

At suitable points in the circuit the two components are added and the product is discharged.

The reaction time which it is possible to attain in any of these systems will be limited by the extent of the cooling surface. For example, system (a) will necessarily give a much slower reaction rate than the other three systems. Another point requiring attention is the pressure drop which will take place in the reactor with a consequent increase in the power requirements.

Thus it can be seen that it is process development by which the essential factors for sulphation are obtained, but it is chemical engineering that specifies the reactor which best suits the task. The final decision will be made taking everything into consideration from an economic viewpoint.

## Fluid-bed technique

This process has been evolved in the petroleum industry for catalytic cracking of oil distillates. Oil vapours are brought into contact with a dense cloud of catalyst particles. This cloud behaves more or less like a liquid. The process requires two reactors: one for cracking the oil and the other in which the catalyst is regenerated.

The fluid process was originally claimed to be any easy method of transporting the catalyst. Whilst this is true, it is also clear that other important advantages can be claimed. Heat transfer between the particles is extremely rapid and this results in the bed temperature being homogeneous. Heat transfer between the gas and the wall of the vessel is greatly increased and there is consequently less risk of local overheating.

One disadvantage of the fluid bed is that the gas phase is also nearly homogeneous, which means that the reactor is only a single-stage mixer. This results in a lower conversion rate than would be obtained under similar conditions with a fixed-bed catalyst. It is important to note that this mixing does not occur in very narrow vessels and care must therefore be taken in the correct design of pilot plant for this operation.

## Hydro-desulphurisation of oils

This process aims at the removal of sulphur from oil by mild and selective hydrogenation. The catalyst which has been evolved by development work is an improved cobalt-molybdenum supported catalyst.

The need to reduce the quantity of hydrogen which is recycled has led to adoption of the 'trickle' technique. Vaporisation of the oil is avoided by allowing it to flow down as a thin liquid film over a fixed catalyst bed. It was found that this led to an additional advantage in that the catalyst activity is not impaired.

It was important, when constructing the pilot plant for this process, to obtain results representative of a commercial-scale reactor. This necessitated a study of the flow over the catalyst bed for varying ratios of catalyst diameter to tube diameter. It was found that when this ratio becomes greater than 1 to 5 more than 30% of the oil flows along the wall of the tube. The pilot plant therefore required the use of small pellets of catalyst.

Even with a catalyst of the right size the flow of liquid is not uniform and it is beneficial to sub-divide the catalyst bed and put liquid-redistributors in between. This also enables cooling of the oil to be carried out between the parts of the catalyst bed in order to compensate for the exothermic reaction which occurs.

**Heating mantles.** Electric surface heating of the *Isomantle* type was introduced in 1947 and is now being applied to vessels, pipelines, valves, processing and many 'special' applications. It is based on the direct heating of the surface concerned. Standard units cater for temperatures up to 400°C. (750°F.) when made of glass yarns and glass wools, but special quartz units are available for temperatures up to 800°C. (1,470°F.). All these units, made by Isopad Ltd., are described in a 16-page illustrated booklet.

**Cyanides in industrial wastes.** Wastes containing cyanides are emitted from many industrial processes, notably metal finishing and coal carbonisation, and the total which could find its way into waterways from these sources is very large indeed. The level of cyanide concentration which can be tolerated in rivers without harm to fish life is about 0.01 p.p.m. (as HCN). Cyanides are removable by normal sewage treatment processes in certain conditions, but wide variations in total concentration may inhibit the biological action of percolating filters, with the result that cyanides pass into the receiving waterway. Therefore, the cyanides present in raw sewage entering a disposal plant should be maintained at as constant a value as possible and preferably below 1 p.p.m., or they should be removed entirely from wastes before they enter the sewer. In any case their complete removal from wastes discharged to rivers is essential. The problem is discussed in an illustrated article by E. A. Whitlock, B.Sc., A.R.I.C., published in pamphlet form by Wallace & Tiernan Ltd. Numerous methods of removing cyanides from wastes are mentioned, but of these there is probably only one—the alkaline chlorination process—by which cyanides may be completely destroyed on a commercial scale at reasonable cost. Practical applications of this process are described.

\*Abstract of a paper presented at a meeting of the Institution of Chemical Engineers in London on March 2, 1954.

# Plant and Equipment

## Switch fuses

A range of heavyweight weather-proof switch fuses for use in rigorous atmospheric conditions has been introduced by the General Electric Co. Ltd. The new fuses comply with Home Office Factory Regulations, I.E.E. Regulations and the relevant British Standards. They are intended for circuits up to 500 volts and are available in 20, 30, 60 and 100 amp. sizes in single pole and neutral, double pole, triple pole, and triple pole and neutral types. They are arranged to take rewirable or H.R.C. cartridge fuses, the carriers for each type being interchangeable.

The heavy cast-iron cases of the new switchgear range are claimed to be completely weatherproof; they are undrilled, with external fixing lugs and gasketed cast-iron end plates. The end plates, which cover slots in the top and bottom of the boxes, can be removed and drilled for conduit entries.

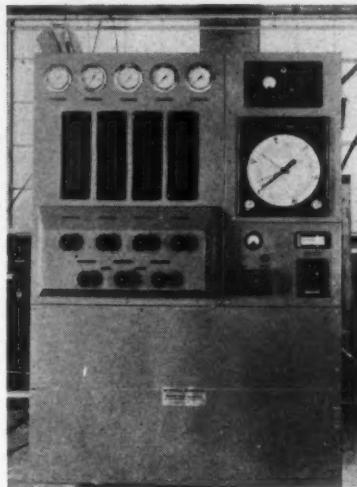
There is generous wiring space inside the cases, so that incoming and outgoing cables may enter and leave by several alternative ways. Wiring and making connections is assisted by the fact that the steel spindle and switch blade assembly are removable merely by axial pressure against a retaining spring. The spring-loaded spindle also ensures correct alignment. Radiused edges on the spindle are said to prevent cracking on the Bakelite insulation when the blades are clamped down.

The connection between the switch and the fuse consists of a solid brass bar, which is the contact on the switch base as well as the bottom contact of the fuse. There is thus no risk of contacts overheating. The switch is interlocked with the case cover which is provided with pillars to accommodate spare H.R.C. cartridges.

The equipment may be used in oil fields, gas works, sugar refineries, chemical works, textile factories and other industries where considerable humidity is encountered.

## Generating nitrogen for metal treatment

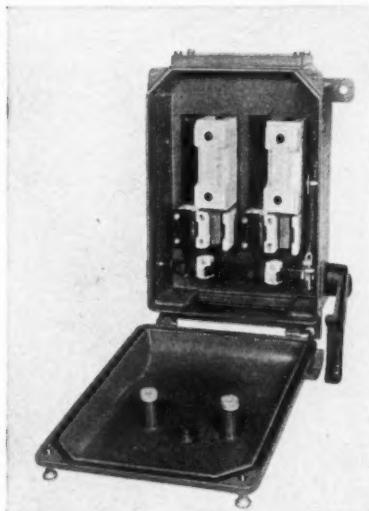
The vast literature on controlled atmospheres for annealing metals testifies to the great industrial importance of the subject. In general, the ideal atmosphere is one which does not react in any way with the metal and, in the case of certain rarer metals which are now becoming of importance, only the truly inert gases, helium, argon, etc., can be used. For the majority of the common metallic materials used in engineering, however, nitrogen also is inert, and this gas is used on a small scale for heat-treatment. The production of oxygen-free nitrogen requires large and expensive plant and, with a few exceptions, the cost of this gas is prohibitive. In any case, an



The 'Nitroneal' generator

inert atmosphere only affords complete protection where there is no possibility of the atmosphere being contaminated either by leakage of air or by evolution of oxygen from refractories or other parts of the vessel in which the heat-treatment is being carried out. For this reason it is desirable to provide in the nitrogen a small percentage of a reducing gas such as hydrogen, to take care of any adventitious contamination, and such mixtures are ideal atmospheres for many metallurgical heat-treatment operations.

Mixtures of hydrogen and nitrogen are, however, used fairly extensively, and a very convenient method of producing such mixtures is by the dissociation of anhydrous ammonia. Ammonia is readily available in a high state of purity and with exceptionally low water content. It liquefies readily on



Weatherproof switchgear

compression and, therefore, a large quantity can be packed and transported in a steel cylinder. Cracked ammonia is widely used in the metallurgical industry, especially where very high purity justifies the expense. The high proportion of hydrogen (75%) puts it in the same category as hydrogen itself, as regards inflammability and reactivity, and its use is, therefore, limited both as regards scale of operation and nature of the work being done. To overcome these drawbacks, and also to reduce the cost, apparatus has been developed and fairly widely used in which the cracked ammonia is burnt with a slight deficiency of air to give nitrogen containing a small percentage of residual hydrogen. Provided precautions are taken to ensure cleanliness of the air, i.e. freedom from obnoxious impurities such as sulphur compounds, etc., and the burnt gas is thoroughly dried, the resulting hydrogen-bearing nitrogen retains substantially the advantages of the original cracked ammonia without its disadvantages. However, the apparatus required is relatively large and requires close control. Its use is, therefore, economical only where a fairly large and continuous output is required.

A recent development, the *Nitroneal* generator, provides the same sort of atmosphere by combining, in one process, the simultaneous cracking and burning of ammonia. The generator employs a platinum metal catalyst through which a controlled mixture of air and ammonia is passed. The catalyst ensures the complete reduction of all the oxygen to water vapour, which is then removed partly by condensation and partly by conventional driers. The dew point of the resulting gas depends solely on the efficiency of drying, and can be reduced to as low as  $-40^{\circ}\text{C}$ . without difficulty and even lower if required. The control panel provides for easy adjustment of the ammonia/air ratio and, subsequently, automatic control ensures constant composition. The hydrogen content of the *Nitroneal* gas can be varied from about 0.25% all the way to 25%, and a built-in analyser indicates and records the hydrogen percentage. The equipment is available in a range of sizes from 100 cu.ft./hr. upwards, and all generators except the 100-cu.ft./hr. model are controlled automatically once the original setting has been made, i.e. any departure of the analysis from the pre-set figure operates the automatic control. Manual control only is provided on the smallest generator in order to keep down the cost and broaden its field of economic usefulness.

A particularly noteworthy feature is the rapidity with which the generator can be brought into service. It is claimed that, from a cold start, a satisfactory atmosphere is produced in about 15 min., and that the composition of the *Nitroneal* gas settles down to the set figure within a further 10 min. at the most. This is of great importance for intermittent use.

## Porous ceramic air slide

During the past few years, the Royal Doulton Potteries have shown on their stand at the B.I.F., in the form of working models, some of the more interesting applications of porous ceramics. These models have been taken from the Royal Doulton development laboratories, where they were designed and built to enable investigations to be made along certain specified lines.

In 1952 was shown a small model air slide, which was followed in 1953 by a fluidised hopper. The principal exhibit this year comprises a combination of these two pieces of equipment on a considerably enlarged scale.

The hopper has a capacity of approximately 60 cu. ft. and feeds an air slide over 30 ft. long. Because of the high conveying rate of the air slide, its capacity has had to be kept down by restricting its effective width to 3 in. To enable the model to operate continuously, the material which is discharged from the air slide is returned to the hopper by means of a bucket elevator. Inspection windows have been provided at strategic points so that the full cycle of operations can readily be observed.

It is virtually impossible to scale up from very small models to full-size plant, primarily because of effects such as that of the hydrostatic head of material in a large hopper, and it is this knowledge which has prompted Doulton to obtain this comparatively large piece of equipment.

In addition, a comprehensive range of porous ceramics for filtrations, dust recovery, diffusion, electrolysis and other processes is being shown.

## Automatic bleed unit

Conventional automatic controllers employing integral action may have some disadvantages when used on continuous or batch processes: an 'overshoot' of the measured variable tends to occur when the variable returns to its desired value after an interruption of the process, which often, in one way or another, renders the control mechanism ineffective. In order to eliminate this 'overshoot,' a British firm has developed a device known as the automatic bleed unit.

This unit is connected to the integral chamber of the controller and the controller nozzle line, so that when an interruption of the process occurs and the control pressure reaches its maximum, a 'bleed' of air from the integral chamber takes place, thus retaining the flapper within its normal working range and keeping the controller mechanism effective. This action will occur continuously until plant conditions return to normal. In consequence, control action begins while the measured variable is returning to its desired value, thereby greatly reducing the possibility of 'overshoot.'

The unit consists essentially of a cast

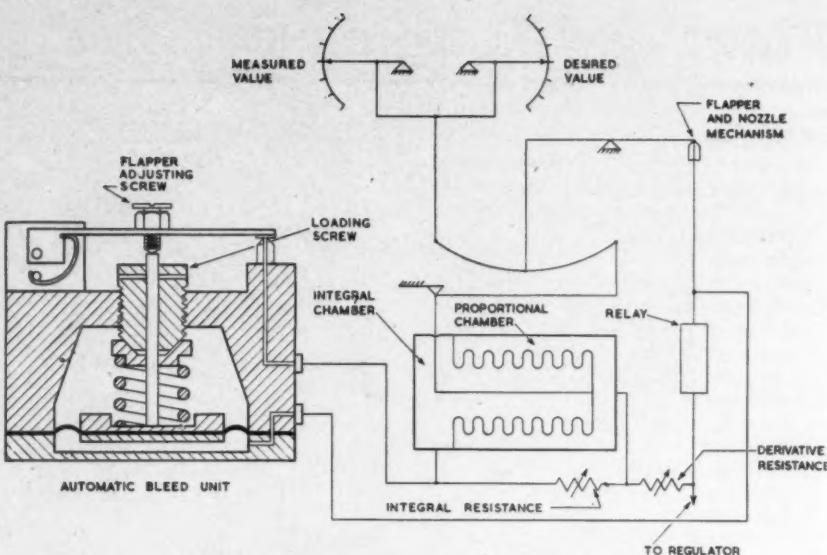


Diagram of the automatic bleed unit used with a conventional automatic controller

body enclosing a flexible diaphragm. This diaphragm is constrained by an adjustable control spring, as shown in the illustration. When the control pressure, which is applied below the flexible diaphragm, reaches its normal maximum and overcomes the control spring, the pivoted arm is raised, opening the nozzle valve connected to the integral chamber of the controller, and bleeding air from the integral chamber to atmosphere. The control spring is adjusted to correspond with the upper limit of the control pressure range by means of the loading screw at the top of the cast body.

Said to be a simple and reliable device, easily added to a standard controller, it in no way detracts from the controller's performance nor modifies its response characteristics.

## Self-cleaning air filter

An automatic self-cleaning air filter, the *Reciprojet*, capable of handling large volumes of heavily dust-laden air, is now being manufactured by Visco Engineering Co. Ltd.

This unit consists of one or more sections of various heights to suit the required capacities, each section comprising a mild-steel frame having two columns of filter cells supported in carrier plates attached to two endless chains. At the base of each section is an oil tank of ample capacity having a large access door.

At predetermined times an electric time switch starts up the fractional horse-power motor which drives the filter columns and an oil-pump motor. This brings the dirtiest cell in each section into the cleaning position, where it is subjected to the scouring of a number of full-bore oil jets which move backwards and forwards across the width of the cell. The dirty cell is initially tilted to allow the oil jets to wash the dirt

forward and downwards through the cell plates, and as the cell continues to move the oil jets effectively scour the front or dirty face.

When the filter columns have been moved by the equivalent of one cell, a limit switch cuts out the filter and oil-pump motors, the time switch then again taking control. All the time the filter is in operation the oil in the system is cleaned by means of an oil strainer unit, the capacity of which is such that all the oil is cleaned at least once between each spraying operation.

The dirty oil remains in the filter tanks in which sludge settles out for eventual removal or, if preferred, it can be drained away to a central settling tank. In this event the oil would be transferred from the settling tank to a clean oil tank ready for the next cleaning operation.

In addition to the greatly improved cleaning effect and the automatic operation of the *Reciprojet*, the filtration efficiency is also considerably increased by the double column of cells through which the dust-laden air has to pass. As the cells in the column on the cleaned air face have been previously in use on the up-stream side of the filter, the oil coating on the cell plates will have dried to some extent and the cells will act as a trap for any oil carry-over from the front and more oily cells. Oil eliminator plates are retained as an additional safeguard against oil carry-over.

## Clearing away water by suction

A new suction drying machine for clearing large quantities of water from outdoor areas and industrial premises such as product-washing plants has now been developed by Cimex-Fraser Tuson Ltd. The unit absorbs and discharges into an emptying tray more than 40 gal. of water in 5 min.

It is driven by a petrol motor and can be fitted with two rollers.

# World News

## GREAT BRITAIN

### Australian contract for Power-Gas

The Power-Gas Corporation Ltd. announce that their Australian subsidiary has secured a contract worth about £1 million, for the construction of chemical plant, in the face of very keen American competition. The contract, for Petroleum & Chemical Corporation (Australia) Ltd., of Sydney, involves the design, manufacture, installation and starting up of a large plant to make high-grade town gas from heavy petroleum oil along with the recovery of valuable by-products. Though the technical planning and plant design will be done at Stockton-on-Tees, much of the bulky fabricated steel equipment will be manufactured in Australia to designs sent out there.

### New office

Honeywell-Brown Ltd. have established an office at 35 Byrom Street, Manchester 3, as an addition to those already existing in London, Glasgow, Birmingham and Sheffield. The new office is under the supervision of Mr. Tom Jackman and will deal with business and enquiries from an area covered by Cumberland, Westmorland, Lancashire, Cheshire and North Wales.

### Expansion at Marconi Instruments

Marconi Instruments Ltd. have greatly increased the productive capacity of their Longacres, St. Albans, works by the opening of a new factory wing. Several sections of the business have been reorganised, permitting a smooth flow of production and distribution at the new level, and a new design centre provides improved facilities for the company's engineers.

A notable feature of the new layout of the works is the strategic placing of the various stores sections, permitting smooth issue of components and raw materials to adjacent assembly departments and the machining sections. In the seven years since the works was centralised at Longacres it has more than trebled in size and, with the new layout, may well be considered a model for efficient production on one floor.

### Three new university Chairs

New chairs of chemical technology and chemical engineering are to be established, two at the University of Nottingham and one at the University of Edinburgh, as follows:

*Nottingham.* The university has accepted the offer of Boots Pure Drug Co. Ltd. to endow two new Chairs: the Lord Trent Chair of Pharmaceutical Chemistry and the Lady Trent Chair of Chemical Engineering, which have a total value of £100,000. The gift commemorates the work that Lord and Lady Trent have done

both for the company and for the City of Nottingham. Lord Trent was chairman of Boots for 26 years and is now the company's first honorary president.

*Edinburgh.* The court of the university has approved the foundation of a Chair of Chemical Technology and the institution of a new degree of Bachelor of Technological Science. This decision has been taken in consultation with the Heriot-Watt College, and the new degree may be later awarded to students in other fields of technology.

### Something from nothing

A new factory employing 500 workers has been opened by W. Edwards & Co. (London) Ltd. at Manor Royal, Crawley, Sussex. The firm was established 35 years ago and today claims to make the most complete range of vacuum equipment in the world. The range falls into four groups: means of producing high vacua (pumps); means for measuring, indicating and controlling high vacuum (instruments); vacuum systems, apparatus and plant (freeze driers, etc.); and accessories (valves, vapour traps, etc.).

Speaking at the official opening of the factory on March 5, Prof. E. N. da C. Andrade, F.R.S., told 300 guests: '... and what is all this vacuum apparatus for?—to produce nothing at all, shut up in a jar. The firm make the finest apparatus in the world for producing as near nothing as possible.'

### New atomic power project for Scotland

With work started on Britain's experimental atomic power station in Cumberland, it has now been decided to build a fast atomic reactor at Dounreay, in Caithness, Scotland. This is expected to show the way to considerable economies in the consumption of uranium.

Some idea of the engineering difficulties involved may be gained from the fact that, for safety, the plant will be housed in a spherical steel shell. Even so, there will be a very remote possibility of a slight leakage of radioactivity should there be a failure in certain parts of the plant. An enterprise of this kind requires a site of 100 acres in open country yet close to sources of labour and with amenities. It also has to be on the coast to facilitate the discharge of effluent and the provision of sea water for cooling.

The new project was announced by the Minister of Works when moving the second reading of the Atomic Energy Authority Bill, which proposes to place the control of atomic energy development in Britain in the hands of a new, semi-industrial body. He said it was necessary to start now to train the men who were going to design, construct and operate power re-

actors for the supply of electricity. British engineering firms must also be ready to take the lead in the world's markets, for undoubtedly, just over the horizon, immense orders were waiting for all kinds of atomic plant.

Some further facts about Britain's atomic power developments were given by Sir John Cockcroft, Director of the Atomic Energy Research Establishment, when he addressed a Parliamentary committee recently. He said that the development period which will be required to gain operating experience of the first atomic power station—using the graphite moderated power unit—and to build and operate a water moderated reactor may easily cover eight years. The efficiency of conversion of heat into electricity would be limited by the present inexperience in operating the uranium fuels and their containing sheaths at high temperatures. If they were limited to 400°C., the efficiency of the conversion would be about 25%. This was about the average of all the British Electricity Authority power stations, but a modern station which operated at higher steam temperatures and pressures had an efficiency of about 33%. If they could develop an improved metal sheath for the uranium fuel and increase the temperature by 100°C., they would do much better and obtain a substantial reduction in the capital cost per unit of power generated.

### Gas industry and coal prices

The gas industry has been impelled to look for sources of supply other than coal—especially oil and natural gas—as a result of the undue proportion of the extra cost of coal which it is having to bear, said the chairman of the Gas Council, Col. Sir Harold Smith, at a luncheon of the Coal Industry Society. Whereas coal prices to the domestic consumer had increased by 160% since 1938, to the gas industry they had increased by 300%.

'If,' said Sir Harold, 'the prices of coal to the gas industry, as compared with the prices for other uses, were back on the 1938 competitive basis, and if ample supplies were available, we should not be so tempted to look for processes which do not require coal. . . . We know of no more efficient way of using coal than by carbonising it and recovering the by-products. How long we can continue to develop our sales of gas and to make more coke available for sale will depend, first, on the availability of the right types of coal and, second, on the price of gas coal as compared with coal supplied for other uses.'

In our January issue (p. 31) we reported a suggestion, made by Dr. F. M. H. Taylor in a paper presented to the Institute of Fuel, that an independent fuel advisory committee should be set up whose main function would be to supervise the price structure of the National Coal Board, and thus fix the prices of the raw material supplied to the gas and electricity industries.

## Symposium on fuel and power in the chemical industry

A symposium on 'Fuel and Power in the Chemical Industry' is to be held by the Graduates and Students Section of the Institution of Chemical Engineers on April 23 at Battersea Polytechnic, London. The morning session (chairman, Prof. T. R. C. Fox) will be concerned with the production of power from fuels, while the afternoon session (chairman, Sir Harold Roxbee Cox) will be devoted to the application of power to the chemical industry and trends in fuel economy.

The programme is as follows:

MORNING SESSION (10 a.m. to 1 p.m.). 'World Energy Resources—Present and Future,' by Dr. A. Parker (D.S.I.R.). *The Production of Power from Fuels: 'Steam Generation for Chemical Plant,'* by R. H. Paddon-Row (Foster Wheeler Ltd.); 'Chemical Engineering Aspects of Gas Manufacture,' by J. J. Priestly (W. C. Holmes & Co. Ltd.); 'The Production of Heavy Fuel Oils,' by Dr. D. P. Plummer (Shell Refining & Marketing Co. Ltd.).

AFTERNOON SESSION (2.15 p.m. to 5.30 p.m.). *The Application of Power to the Chemical Industry:* 'The Use of Electricity in the Electro-Chemical Industry,' by R. S. Clegg (The British Aluminium Co. Ltd.); 'The Use of Steam in Evaporation,' by B. N. Reavell (Kestner Evaporator & Engineering Co. Ltd.); 'The Use of Gas in the Chemical Industry,' by R. F. Hayman (Gas Council); 'The Burning of Heavy Fuel Oils,' by D. A. Monk (Peabody Ltd.). *Trends in Fuel Economy:* Prof. R. J. Sargent.

There will be approximately 15 min. for discussion at the end of each paper.

A summing-up will be given by Prof. F. H. Garner, Director of the Department of Chemical Engineering, University of Birmingham.

## Cellulose chemistry and the cellulose industry

The Scottish Sections of the Royal Institute of Chemistry have arranged a symposium on cellulose to be held at the University of St. Andrews from Monday, June 28, to Friday, July 2 (inclusive).

Lectures and discussions will be grouped as follows: (1) 'The Chemistry of Cellulose and its Derivatives'; (2) 'Paper and Pulp'; and (3) 'Industrial Applications of Cellulose and its Derivatives.'

The speakers will include Dr. D. W. Hill and Dr. A. R. Urquhart (British Cotton Industry Research Association), Dr. J. L. Bolland (British Rayon Research Association), Mr. H. A. Thomas (Courtaulds Ltd.), Dr. W. A. Caldwell and Dr. A. W. Baldwin (Imperial Chemical Industries Ltd.), Dr. I. Jullander (Mo och Domsjo A/B., Sweden) and Mr. L. G. Cottrell (Albert E. Reed & Co. Ltd.).

Lectures and discussions will be held during the mornings. Informal meetings, works visits and social events will be

arranged for the afternoons. There will be a symposium dinner.

Registration forms may be obtained from the assistant secretary, Royal Institute of Chemistry, 30 Russell Square, London, W.C.1. As accommodation is limited, priority will be given to visitors from overseas and to members of the Royal Institute of Chemistry who make application by May 1.

## Change of address

The London office of Joseph Crosfield & Sons Ltd. is now at Hesketh House, Portman Square, London, W.1 (telephone: Welbeck 4466).

## I.C.I., Dunlop and Monsanto to make synthetic rubber

With large-scale production facilities being planned by I.C.I., Monsanto, and the Dunlop Rubber Co., Britain will at last have a synthetic rubber industry. The three companies disclosed their plans last month within a few days of each other.

I.C.I. should be making pilot-plant quantities available later this year; the full-scale plant at Wilton, North Yorkshire, with a planned capacity of about 10,000 tons p.a., will probably not be in operation before 1956. Using butadiene produced in I.C.I.'s oil cracker at Wilton, it is intended to make copolymers, using a variety of second components including acrylonitrile and styrene. These copolymers will range from oil-resisting substances of the *Perbunan* type to hard, reinforcing resins. The resins will be particularly attractive for use in shoe-lining compositions, and they will also be available in the form of latices suitable for the production of emulsion paints. Some of the polymers to be made will be similar to those already available in the U.S., but others will be novel, both in composition and in properties. They will all be speciality materials designed to provide effects which at present can only be achieved by restricted imports from the U.S.

Monsanto Chemicals Ltd. have recently completed a tonnage-scale pilot plant from which a range of styrene-butadiene rubber-like resins will be available from July this year. About £500,000 is also to be spent on a large-scale plant which will begin production in about two years with a planned initial capacity of 4,000 tons p.a.

Dunlop's project will also cost £500,000 and will produce enough synthetic rubber to experiment with tyres and other products. Work has already begun on the site at Fort Dunlop. The company have for some time been producing pilot quantities of synthetic rubbers for a wide range of applications.

## GREECE

### Trade agreement with Austria

Included in the goods to be exported to Austria under a new trade agreement are turpentine oil, colophony, stearine and other fatty acids, chrome ore, barytes and white bentonite. Among the goods to be imported from Austria in exchange are various industrial felts, especially felts for paper making, special taps and valves, pumps, compressors, exhausters, glass perforators and other glass-making machinery and industrial furnaces.

## NORWAY

### Big aluminium plant starts production

Norway's biggest aluminium plant, at Sunndalsöra, West Norway, is starting production this month, but it is not expected that full production will be achieved until next year, owing to delays in completion of the Aura hydroelectric plant which will supply the necessary power.

The Sunndalsöra plant will produce about 40,000 tons p.a. of aluminium, and will therefore almost double the present total Norwegian aluminium output which is about 52,000 tons p.a. Much of the £12½ million which the plant is estimated to cost has been provided by an American E.C.A. loan which is to be repaid in the form of aluminium deliveries. Director-General Aage W. Owe estimates that, at the present price for aluminium on the New York commodity market, it will be necessary to deliver about 65,000 tons of aluminium over a period of 10 years to repay the loan. The loan carries interest at 2½%.

## FRANCE

### New solar still

An apparatus has been invented by a French engineer for the distillation of sea water and recovery of moisture in the air by solar energy. The pilot apparatus set up has a surface of only 8 sq. m. for catching the sun's rays, but this enables it, even in winter, to utilise more than 17,000 kw./hr. of energy, it is stated.

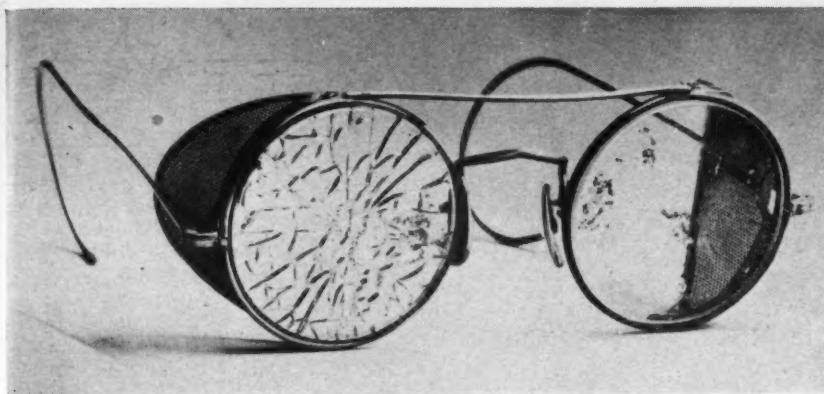
Salt and magnesium can also be recovered by means of the still, which is claimed to be very economical.

### Oil refinery instrumentation contract

The French Société C.O.M.S.I.P. of Croissy-sur-Seine have secured the contract for the instrumentation of the extension of the Lisbon oil refinery of S.A.C.O.R. (Sociedade Concessionaria da Refinacao de Petroleos em Portugal)—a project covered by the Six-Year Plan but financed by the company's own resources. A U.K. firm had also tendered. The work will, it is reported from Paris, cost 200 million francs and take eight months. It comprises control apparatus and automatic regulations for topping, cracking, catalytic polymerisation units, gas plant, petrol treatment, asphalt blowing, distillation, and steam, air and water supplies.

### Lille trade fair

The Lille International Trade Fair, to be held from May 1-16, is a technical and economic exhibition open to industries all over the world. It is situated in the heart of the northern region of France, one of the most important agricultural and industrial areas in the northern countries. It is one hour by road from Brussels, less than 250 km. from London and Luxembourg, 300 km. from The Hague and 550 km. from Frankfort.



#### GOOGLES STOP FLYING METAL

Under the Iron & Steel Foundries Regulations, 1953 (No. 1464), which came into force on January 1, 1954, certain foundry workers must be provided with suitable eye protection. The wisdom of these regulations is illustrated in the above photograph. The goggles shown, made by J. & R. Fleming Ltd. and fitted with 'Armorglas' toughened lenses, were being worn by a man engaged in tapping metal from a cupola. Flying metal from the tapping hole struck the goggles; it is possible to see the metal still adhering to the lenses. The heat and the impact were sufficient to fracture the right-hand lens, but no fragments were displaced and the wearer was uninjured. Had he not been wearing suitable eye protection, he might well have lost at least one eye.

The Fair occupies an area of about 240,000 sq. yd., of which 96,000 sq. yd. include large covered halls. The Grand Palace itself covers about 1 hectare. The frontage measures 120 m. long by 30 m. high, the whole entirely covered with aluminium.

About 3,500 exhibitors participate every year and include firms of about 15 different nationalities. One and a half million visitors are admitted annually, of which 300,000 are Belgians, Dutch, Luxemburgers, English and Germans. Fifteen different countries allow special facilities and reductions in railway fares, aerial and sea transport upon presentation of the 'Fair Identity Card' issued by the Fair authorities, French Consulates and the principal travel agencies the world over.

#### ITALY

##### Methane use grows

Sales of methane during 1953 reached 2 milliard cu. m. This is the equivalent of 3½ million tons of coal—a third of the national requirements—which would cost 40 milliard lire to import. The volume of methane used continues to rise and now averages 8½ million cu. m. a day. By the end of 1954 it is planned that all the cities of northern Italy should be joined by a network of pipelines.

#### GERMANY

##### Synthetic rubber plans

Some important West German tyre manufacturers plan to set up a plant for the manufacture of buna rubber with an annual production capacity of 30,000 tons, according to the Association of the West German Chemical Industry. The project, supported by the West German Government, was intended to be carried out by the Chemische Werke Hüls, at present West Germany's sole manufacturer of

buna. The Chemische Werke were turning out buna at a monthly rate of 300 tons.

It was also intended to abandon the present costly acetylene production method, which used coal as basic material, and to introduce a new process using alcohol instead (see *CHEMICAL & PROCESS ENGINEERING*, 1954, 35 (1), 32).

#### TURKEY

##### Germans, French and Italians share cement plant contracts

Protocols relating to the construction of 20 cement plants have been signed in Ankara. Seven contracts were awarded to the French firm of Fives (Lille); twelve to German firms, of which six went to Krupp, two to Humboldt, two to M.I.A.G. and two to Polysius; and one to the Italian firm of Ansaldo. Terms were reported to be 20% before erection and the balance over five years at 4% interest. When all these plants are in operation it is estimated that Turkish annual production will approximate 3.3 million tons.

##### Sugar industry's expansion plans

Seven new sugar factories are to be built at Kayseri, Erzurum, Susurluk, Burdur, Malatya, Erzincan and Elazig. Three contracts were awarded to two French firms and the four others to two German firms, bringing to eleven the total number of sugar factories now under construction. When all are in production there should be a total sugar output of 375,000 tons.

#### NETHERLANDS

##### 'Terylene' production licensed

Imperial Chemical Industries Ltd. have concluded a licence agreement with Algemeene Kunstzijde Unie N.V., Arnhem, the Dutch manufacturers of rayon and nylon, under which the latter company will have the right to manufacture in the Nether-

lands the Belgium the new polyester fibre, *Terylene*.

In addition to production by I.C.I. at the great factory now being built at Wilton, North Yorkshire, this fibre will be produced in France, Italy and Western Germany and also in Canada where I.C.I. of Canada Ltd. are building their own plant for the production of the fibre at a cost of more than \$20 million.

#### WEST INDIES

##### Sulphur exports to U.K.

The first shipment of 800 tons of sulphur manufactured locally by Trinidad Leaseholds (Chemical) Ltd. was recently shipped to the U.K. It is expected that, after taking care of local requirements, about 5,000 tons will be exported annually.

#### VENEZUELA

##### Oxygen plant

A factory which has been erected by an Italian syndicate commenced production of oxygen in January. In the near future this plant will also produce carbide.

##### Sulphur production plans

The Venezuelan Sulphur Corporation expect to start production of sulphur early in 1954 at an initial annual rate of 200,000 tons. The company is seeking a loan of Bs.20 million in the U.S.A. The Venezuelan Sulphur Corporation have the El Pilar deposits, located some 10 miles south of Carupano. These are altogether separate from the recently discovered deposits at Puerto Piritu, where future plans are uncertain and where it is known that British investment would be welcomed.

#### PHILIPPINES

##### New paper projects

A plan formulated for a Philippine pulp and paper project envisages a yearly (300 days) output of 61,500 tons of paper from Philippine hardwoods. Production would include 16,500 tons of kraft linerboard, 14,500 tons of kraft bag and wrapping paper, 18,000 tons of newsprint and 12,500 tons of printing and writing papers.

Experimental work at several U.S. laboratories disclosed that, in general, Philippine hardwoods are short fibred. It was found, however, that the addition of long-fibred stock, such as abaca and kenaf, in amounts up to 20% would improve most papers produced from the woods tested to approximate U.S. standards. For newsprint, the addition of clay to the raw-material mixture, 60% groundwood and 40% chemical pulp, upgraded the end product to standard quality.

Commercial production of paper pulp from indigenous raw materials has barely been initiated in the Philippines and newsprint production is still in the planning stage. Paper requirements, difficult to estimate because of restricted imports and inadequate statistics, are believed to approximate 90,000 tons p.a. Local production in 1953 probably did not exceed 11,500 tons.

## CANADA

### Nickel ore plant enlarged

The capacity of the concentrator at the Creighton mine of the International Nickel Co. of Canada Ltd. has been increased by 20% to 12,000 short tons/day of ore. The mill building has been enlarged to a length of 465 ft. It is 175 ft. wide and 70 ft. high. Two crushers have been added to the original four. Thirty-six flotation machines were also added, bringing the total to 180.

The capacity of the mill, in which nickel-copper ore is separated from rock before smelting, has been enlarged twice in response to increased nickel demand for defence and civilian requirements. When it was designed in 1948, it was to have a daily capacity of 6,000 tons, but plans were changed in 1950—two weeks after the outbreak of hostilities in Korea—to enable the mill to accommodate 10,000 tons/day of ore. The new capacity of 12,000 tons is, therefore, double that which was originally planned. (All tonnages are in short tons.) International Nickel is at present producing refined nickel at an annual rate of approximately 275 million lb.

The concentrator is at the site of Creighton No. 7 shaft, completed in 1951 at one of the company's oldest operating mines—Creighton, which has a record of production dating back to 1900. The shaft brought to 14 the number in operation at the company's mines. The Creighton concentrator is a compact, streamlined plant with unusual features of design and operation. The head frame and hoist house are integral parts of the mill building and ore from the mine is hoisted directly into the crushing plant. Part of the mill feed is carried by conveyor from another Creighton shaft two-thirds of a mile away. The grinding mills are among the largest in the world. The plant's water supply is obtained through a 6-mile pipeline, and the bulk concentrate—a thin, mud-like mixture—which comes from the mill is pumped through another pipeline to Inco's reduction plants at Copper Cliff  $7\frac{1}{2}$  m. away.

Since 1949—the year before the Korean conflict began—the nickel industry of the free world has increased its annual production from about 265 million lb. to some 340 million lb., or by over 25%. Almost half of the 1953 production was allocated for military, atomic energy and stockpile requirements. Over 65% went to the United States for both defence and civilian purposes. This was stated by the chairman of International Nickel Co. of Canada Ltd. in his annual review. He said that the further increase now achieved in International Nickel's production capacity and other expansion programmes under way indicate that free world nickel production should reach about 385 million lb. in 1954. Based upon announced expansion programmes, the free world capacity by 1960 should be at least 75% greater than that existing in 1949.

### The Leonard Hill Technical Group—April

**Manufacturing Chemist**—Lilac Perfumes; Organic Compounds of Sulphur; Chemotherapy and Selective Toxicity, Part 3; Paper Chromatography and Electrophoresis; Anti-polio Vaccine; Heat Transfer Problem in Metal Plant; Progress Reports: Chemotherapy, Fertilisers and Plant Nutrients.

**Food Manufacture**—Dublin Quick-freezing Factory; The Vitamins, Part 2; Beet Sugar Refining in the U.S.A., Part 2; Hygiene in Meat Manufacture.

**Fibres**—Testing and Measuring Instruments for Wool; Instruments used in the Paper Mill Laboratory; Measuring the Wear Resistance of Textile Fabrics; Testing the Fastness of Coloured Fabrics to Light and Washing; Measurement of Crimp in Wool.

**Paint Manufacture**—O.C.C.A. Exhibition Preview; Alkali-induced Isomerisation of Linseed Oil; Printing Inks for the Electronic Industry; Dispersed Pigments; Protection of Ferrous Metals—Battersea Exhibition; Organic Protective Coatings.

**World Crops**—Economics of Peasant Cane Farming in Trinidad; Fertiliser Plant in Mississippi; Methyl Bromide as a Fumigant in Cargo Ships; Handling Bananas at the Port of Bristol.

**Petroleum**—Simple Corrosion Theory as an Aid to Material Selection, Part 1; The Choice of Lubricants; Operational Research.

**Atomics**—The Counting of Low-level Activities; Portable Gamma-radiation Beacon; Nuclear Reactors and Power Production, Part 1.

**Building Materials Digest**—Preservative Treatment of Timber, Part 2; Copper and its Functions in Building; Gypsum Building Products.

**Muck Shifter**—Grand Contour Canal, Part 3; Poland's System of Waterways; Construction of the Palisades Dam, U.S.A.

## SOUTH AFRICA

### Calcium cyanamide project

Construction of a calcium cyanamide plant for South African Cyanamid (Pty.) Ltd. is starting at Witbank. The plant is expected to be completed early in 1955. The calcium cyanamide will be used as raw material in the large cyanide-producing plant which the company has operated at Witbank since 1938. At present, this intermediate is imported from the firm's parent company in Canada, North American Cyanamid Ltd.

The new plant has been designed to produce enough calcium cyanamide to meet the demand for *Aero* brand cyanide, with enough left over for specialised uses such as in agriculture. The plant will embody an expansion potential sufficient to meet foreseeable demand.

Agreement has been reached with Rand Carbide Ltd. for long-term supplies of calcium carbide, the principal raw material.

### Duty on process plant

Considerable difficulty is experienced by South African Customs officials in respect of shipments of goods ostensibly classifiable under Tariff Items 88, 118 (b) or (g), 119 (a) and 107, but in respect of which the question arises as to the admissibility under those tariffs of items such as structural steelwork, tanks, etc. The main difficulty lies in incorrect or incomplete entry of such shipments by importers/clearing agents because of the lack on suppliers' invoices of detailed particulars (*i.e.* the description, value, etc.) in respect of the steelwork, tanks, etc. Information of this nature should be readily available, as orders for 'fixed plant' and the like are preceded usually by lengthy negotiations and/or the overseas supplier is furnished with detailed specifications, and there would appear to be no reason why South African importers/manufacturers' representatives when placing relative orders should not instruct the overseas supplier in regard to entry particulars required in the Union and at the same time stipulate that a detailed specification be attached to the invoice and that the latter document must reflect separate values for items such as structural steelwork, tanks, etc.

The tariff items enumerated above relate to boilers and boilerhouse plant, fixed plant and machinery for factory installation, other machinery, etc., for manufacturing and industrial purposes, machinery, etc., used in connection with the generation, storage, transmission, distribution of, and lighting by, gas or electric power, and water filtering plant.

The U.K. Senior Trade Commissioner reports that lengthy delays in clearing goods through Customs often occur, particularly when composite plant is imported and some of its parts which are not separately invoiced (*e.g.* tanks, steelwork, pumps, valves) have to be cleared under other tariff items and entered at higher rates of duty.

## INDIA

### Newsprint project's progress

The revised cost of India's first newsprint mill at Napanagar (Madhya Pradesh State, formerly Central Province and Berar) is now estimated at 54.8 million rupees, as against the previous estimate of 60 million rupees. This figure has been arrived at after the scrutiny of the project by a three-man expert committee appointed by the Indian Government last year, and a further examination of the committee's suggestions by the project officials.

The mill, which had received loans from the State Government totalling 30 million rupees till the end of 1953, needs further financial assistance (about 12 million rupees) to go into full production. The total cost of plant and machinery is about 21.5 million rupees, which only equipment worth 1.3 million rupees remains to be ordered.

The erection of the mill's chemical plant is expected to be completed in January 1955. Meanwhile, the mill is to go into production with imported chemical pulp and the trial runs are likely to start in July this year. The rated capacity of the mill is 100 tons/day or 30,000 tons p.a.

## UNITED STATES

### Chemical Engineers' Institute form nuclear division

The American Institute of Chemical Engineers is to form a Nuclear Engineering Division which will be composed of scientists from practically all technical organisations engaged in atomic energy work. A primary objective of the new division will be the advancement of peaceful uses of the atom. Plans have already been set for an exchange of non-military information at a five-day Nuclear Engineering Congress to be held in conjunction with the University of Michigan in Ann Arbor, Michigan, on June 20 this year. Belgium, Canada, France, India, Italy, the Netherlands, Norway, Spain, Sweden, the United Kingdom and possibly several other countries will be represented.

### Monsanto Chemicals' investment plans

The Monsanto Chemical Co. has announced that approval has been given to projects this year involving an expenditure of \$41 million.

About the largest new project started by the company last year is the polythene plant at Texas City, which is expected to be in production early in 1955. With this addition, Monsanto will become the first American firm to make the following six major plastics: styrene, phenolics, cellulose, vinyls, amino plastics and polythene.

Plastics and synthetic resins are the company's largest market. They accounted for 14% of total record 1953 sales of \$340,616,526 in 1953, about 28% more than in 1952.

### Polythene from fuel oil

Allied Chemical & Dye Corporation, New York, has opened its new Niagara River petrochemical plant at Tonawanda, N.Y., for production of polythene products. The ethylene is derived from gas which is produced from fuel oil in equipment designed and erected by Semet-Solvay Engineering Division. This is an innovation in the petrochemical industry, as most plants have been built near natural-gas lines. Production is estimated at 20 million lb. p.a.

### Titanium, nitrogen and cement targets to be raised

The United States Government has decided that domestic production of titanium, Portland cement and nitrogen should be increased. An announcement by the Office of Defence Mobilisation stated that production goals for the three materials had been expanded as follows:

**Titanium** production to be raised to a total of 37,500 short tons annually by 1956.

**Portland cement** output to be expanded to a total of 265½ million barrels annually by the end of 1954.

**Nitrogen** production to be raised to 3½



SHIPMENT OF BRITISH TECHNICAL BOOKS FOR U.S.A.

In view of the recent somewhat adverse criticism of British technical books, expressed by the daily Press, the above picture is perhaps of more than usual interest. The chairman, Mr. W. Leonard Hill (left), and the export manager, Mr. R. Thixton, of Leonard Hill Ltd., had the unusual pleasure of being able to take the author of one of the books they publish to the Victoria Docks, London, recently to show him a shipment of his book on its way to the United States. Not all the consignment is seen here, and while this single order may not be a record, it is certainly an unusually large order for one particular book.

Mr. Edward Gurr, F.R.I.C., F.R.M.S., F.L.S., the author of the book 'A Practical Manual of Medical and Biological Staining Techniques,' who is in the centre of the photo, is one of the world's leading authorities on biological stains and reagents. He is the founder and managing director of Edward Gurr Ltd., of East Sheen, London, the makers of 'Michrome' biological stains and reagents. These highly specialised products, which are exported to all countries of the world, are essential for the study and control of diseases in humans, animals and crops.

million short tons of contained nitrogen by 1957. The previous goal was 2,930,000 tons by 1955.

To meet these expanded goals, the O.D.M. said that it would grant tax benefits such as fast depreciation to companies willing to expand production of the materials in question.

## TUNISIA

### Natural gas found

Natural gas discovered at Abderrahmane, in the Cape Bon region, is expected to be available for consumption in Tunis by the summer of 1954. The construction of the pipeline will be started soon, and when completed it will supply the gas works in Tunis, which will cease to manufacture coal gas while the deposit of petroleum gas holds out.

## COLOMBIA

### Petroleum projects

The new oil refinery on the Caribbean coast is to be built by the International Petroleum Co. of Colombia, probably at Cartagena, where the Andean pipeline debouches. Its estimated cost is 100 million pesos. The main conditions of the contract are that the refinery should have a capacity of not less than 25,000 barrels/day, that it should be in operation by the last quarter of 1957, that it should refine primarily Colombian crude and that its products should be destined primarily for sale in Colombia on the open market without special or preferential prices other than those established by the Government.

The Colombian Petroleum Co. have placed in operation at Tibu, in the Barca Concession, two new units planned to satisfy the demand for refined products in the province of North Santander. They are: a plant designed and constructed by the Fluor Corporation to convert 61 million cu.ft./day of gas into 61,000 gal. of gasoline by an absorption process; and a topping plant to produce gasoline, kerosine and diesel fuel from 2,200 barrels/day of crude. The designs provide for the later production of propane gas for domestic use in Cucuta, and the conversion of the topping plant to the production of gasoline only, if necessary.

## BRITISH HONDURAS

### Tannic acid plant expansion

Plans are afoot to expand the production of the tannic acid factory at Corozal, and up-to-date machinery is being installed so that the product can be made in a concentrated powder form instead of liquid in drums as at present. It is believed that there is ample mangrove bark available locally to keep the factory going to full capacity.

## CHILE

### Acid plant to be imported

The Sali Hochschild Mining Co. has received a favourable reply from the Chilean Ministry of Mines to its request for a duty-free import of equipment for a complete sulphuric acid plant, 20 metric tons daily capacity, to be installed at Copiapo.

### Sulphur exports to Europe

The Chilean National Council of Foreign Commerce has authorised the export of 60,000 metric tons of sulphur to Britain, West Germany and France. Bids of between 32.10 and 32.90 U.S. dollars per metric ton c.i.f. have been accepted from these countries for 20,000 tons each. Delivery will be made partly from stocks (20,000 tons are ready at Antofagasta) and the remainder from production during the next few months. Foreign exchange accruing as a result of these sales will be used partly for the import of vehicles.



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